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2,2-Dichloro-*N*-(3,4-dimethylphenyl)-acetamide

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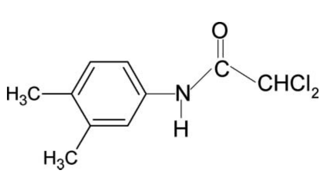
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.005 Å; R factor = 0.056; wR factor = 0.187; data-to-parameter ratio = 15.4.

In the title compound, $C_{10}H_{11}Cl_2NO$, the N—H bond is *syn* to the 3-methyl substituent in the aromatic ring, similar to that observed in *N*-(3,4-dimethylphenyl)acetamide and to the 3-chloro substituent in 2,2-dichloro-*N*-(3,4-dichlorophenyl)-acetamide, and contrasting with the *anti* conformation observed for the 3-methyl substituent in 2,2,2-trichloro-*N*-(3,4-dimethylphenyl)acetamide. On the other hand, it is *anti* to the C=O bond. An intermolecular N—H···O hydrogen bond links molecules into infinite chains along the b axis.

Related literature

For the preparation of the compound, see: Shilpa & Gowda (2007). For related structures, see: Gowda *et al.* (2007, 2008, 2009)



Experimental

Crystal data

$C_{10}H_{11}Cl_2NO$
M_r = 232.10
Monoclinic, $P2_1/c$
a = 11.951 (1) Å
b = 10.534 (1) Å
c = 9.303 (1) Å
β = 111.26 (1)°
V = 1091.5 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.56 mm^{-1}
T = 299 K
0.28 × 0.20 × 0.12 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2007)
T_{min} = 0.859, T_{max} = 0.936
4567 measured reflections
2214 independent reflections
1495 reflections with $I > 2\sigma(I)$
R_{int} = 0.020

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.187
S = 1.20
2214 reflections
144 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1^{i}	0.84 (4)	2.07 (4)	2.894 (3)	166 (3)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2269).

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supplementary materials

Acta Cryst. (2009). E**65**, o1607 [doi:10.1107/S1600536809022557]

2,2-Dichloro-*N*-(3,4-dimethylphenyl)acetamide

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Comment

As part of a study of the effect of ring and side chain substitutions on the crystal structures of aromatic amides (Gowda *et al.*, 2007; 2008; 2009), the structure of 2,2-dichloro-*N*-(3,4-dimethylphenyl)acetamide (I) has been determined. The conformation of the N—H bond in the title compound is *syn* to the 3-methyl substituent in the aromatic ring [similar to that observed in *N*-(3,4-dimethylphenyl)acetamide (Gowda *et al.*, 2008) and to the 3-chloro substituent in 2,2-dichloro-*N*- (3,4-dichlorophenyl)-acetamide (Gowda *et al.*, 2007)], and contrasting the *anti* conformation observed for the 3-methyl substituent in 2,2,2-trichloro-*N*-(3,4-dimethylphenyl)acetamide (Gowda *et al.*, 2009). On the other hand, it is anti to the C=O bond, as observed in other amides. A N—H···O intermolecular hydrogen bond links molecules into infinite chains along the *b* axis. (Table 1, Fig. 2).

Experimental

Compound (I) was prepared and characterized according to the literature method (Shilpa and Gowda, 2007). Single crystals were obtained from the slow evaporation of an ethanolic solution of (I).

Refinement

The H atoms of the methyl groups were positioned with idealized geometry using a riding model [C—H = 0.96 Å]. The other H atoms were located in difference map and their positional parameters were refined freely [N—H = 0.84 (4) Å and C—H = 0.87 (4)–0.97 (4) Å]. All H atoms were refined with isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

Figures

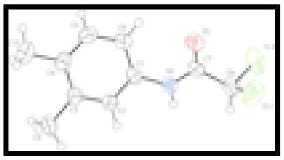

Fig. 1. Molecular structure of (I), showing the atom labelling scheme. The displacement ellipsoids are drawn at the 50% probability level.

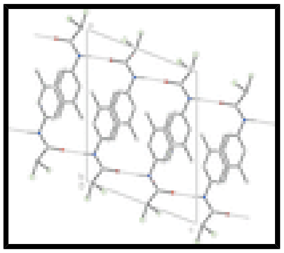

Fig. 2. Molecular packing of (I) with hydrogen bonding shown as dashed lines.

2,2-Dichloro-*N*-(3,4-dimethylphenyl)acetamide

Crystal data

$C_{10}H_{11}Cl_2NO$	F_{000} = 480
M_r = 232.10	D_x = 1.412 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1463 reflections
a = 11.951 (1) Å	θ = 2.6–27.9º
b = 10.534 (1) Å	μ = 0.56 mm^{-1}
c = 9.303 (1) Å	T = 299 K
β = 111.26 (1)º	Prism, colourless
V = 1091.5 (2) Å^3	0.28 × 0.20 × 0.12 mm
Z = 4	

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	2214 independent reflections
Radiation source: fine-focus sealed tube	1495 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.020
T = 299 K	θ_{max} = 26.4º
Rotation method data acquisition using ω and φ scans	θ_{min} = 2.7º
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2007)	$h = -14 \rightarrow 13$
T_{min} = 0.859, T_{max} = 0.936	$k = -13 \rightarrow 6$
4567 measured reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.056	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.187	$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.20	$(\Delta/\sigma)_{max}$ = 0.023
2214 reflections	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
144 parameters	$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Experimental. CrysAlis RED (Oxford Diffraction, 2007) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Cl1	−0.02466 (12)	0.39913 (11)	0.15433 (13)	0.0798 (4)
Cl2	−0.11319 (10)	0.15743 (13)	0.00649 (16)	0.0885 (5)
O1	0.1417 (2)	0.1762 (2)	0.2797 (2)	0.0521 (7)
N1	0.1947 (2)	0.1901 (3)	0.0686 (3)	0.0389 (6)
H1N	0.173 (3)	0.217 (3)	−0.023 (4)	0.047*
C1	0.3132 (3)	0.1393 (3)	0.1286 (3)	0.0360 (7)
C2	0.3945 (3)	0.1829 (3)	0.0644 (3)	0.0383 (7)
H2	0.370 (3)	0.240 (3)	−0.020 (4)	0.046*
C3	0.5126 (3)	0.1407 (3)	0.1172 (4)	0.0400 (7)
C4	0.5503 (3)	0.0526 (3)	0.2372 (4)	0.0442 (8)
C5	0.4666 (3)	0.0078 (3)	0.2959 (4)	0.0493 (9)
H5	0.487 (3)	−0.050 (4)	0.367 (4)	0.059*
C6	0.3489 (3)	0.0492 (3)	0.2448 (4)	0.0439 (8)
H6	0.295 (3)	0.013 (3)	0.284 (4)	0.053*
C7	0.1210 (3)	0.2076 (3)	0.1455 (3)	0.0384 (7)
C8	0.0029 (3)	0.2709 (4)	0.0500 (4)	0.0481 (8)
H8	−0.003 (3)	0.302 (4)	−0.051 (4)	0.058*
C9	0.5985 (3)	0.1928 (4)	0.0464 (5)	0.0580 (10)
H9A	0.6372	0.1238	0.0153	0.070*
H9B	0.6580	0.2442	0.1209	0.070*
H9C	0.5550	0.2435	−0.0420	0.070*
C10	0.6786 (3)	0.0066 (4)	0.3023 (5)	0.0664 (11)
H10A	0.7313	0.0774	0.3425	0.080*
H10B	0.6986	−0.0336	0.2221	0.080*
H10C	0.6876	−0.0532	0.3835	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0971 (9)	0.0780 (8)	0.0699 (7)	0.0313 (6)	0.0370 (6)	−0.0031 (5)

Cl2	0.0435 (6)	0.1112 (10)	0.1088 (10)	−0.0225 (6)	0.0251 (6)	−0.0137 (8)
O1	0.0568 (15)	0.0711 (16)	0.0349 (12)	0.0059 (12)	0.0246 (11)	0.0034 (11)
N1	0.0367 (14)	0.0520 (16)	0.0303 (13)	−0.0016 (12)	0.0147 (11)	0.0027 (12)
C1	0.0375 (16)	0.0395 (16)	0.0341 (15)	−0.0027 (13)	0.0167 (13)	−0.0061 (13)
C2	0.0411 (18)	0.0404 (17)	0.0348 (15)	0.0007 (13)	0.0154 (14)	0.0031 (13)
C3	0.0377 (17)	0.0404 (17)	0.0442 (17)	0.0008 (13)	0.0174 (15)	−0.0070 (14)
C4	0.0420 (18)	0.0405 (17)	0.0447 (17)	0.0050 (14)	0.0092 (15)	−0.0068 (15)
C5	0.061 (2)	0.0444 (19)	0.0425 (18)	0.0087 (17)	0.0188 (17)	0.0064 (16)
C6	0.051 (2)	0.0427 (18)	0.0412 (17)	−0.0029 (15)	0.0208 (15)	0.0018 (15)
C7	0.0385 (17)	0.0451 (17)	0.0352 (16)	−0.0079 (14)	0.0176 (13)	−0.0056 (14)
C8	0.0407 (18)	0.065 (2)	0.0426 (18)	−0.0003 (16)	0.0199 (15)	−0.0017 (17)
C9	0.044 (2)	0.059 (2)	0.082 (3)	0.0034 (16)	0.036 (2)	0.0025 (19)
C10	0.052 (2)	0.068 (3)	0.069 (2)	0.0146 (19)	0.011 (2)	0.001 (2)

Geometric parameters (Å, °)

Cl1—C8	1.763 (4)	C4—C10	1.510 (5)
Cl2—C8	1.763 (4)	C5—C6	1.382 (5)
O1—C7	1.227 (4)	C5—H5	0.87 (4)
N1—C7	1.334 (4)	C6—H6	0.93 (4)
N1—C1	1.425 (4)	C7—C8	1.522 (5)
N1—H1N	0.84 (4)	C8—H8	0.97 (4)
C1—C6	1.384 (4)	C9—H9A	0.9600
C1—C2	1.390 (4)	C9—H9B	0.9600
C2—C3	1.389 (4)	C9—H9C	0.9600
C2—H2	0.95 (3)	C10—H10A	0.9600
C3—C4	1.395 (5)	C10—H10B	0.9600
C3—C9	1.510 (5)	C10—H10C	0.9600
C4—C5	1.385 (5)		
C7—N1—C1	126.7 (3)	O1—C7—N1	125.4 (3)
C7—N1—H1N	118 (2)	O1—C7—C8	121.0 (3)
C1—N1—H1N	115 (2)	N1—C7—C8	113.6 (3)
C6—C1—C2	119.8 (3)	C7—C8—Cl1	109.5 (2)
C6—C1—N1	123.0 (3)	C7—C8—Cl2	108.9 (3)
C2—C1—N1	117.2 (3)	Cl1—C8—Cl2	110.92 (18)
C3—C2—C1	121.4 (3)	C7—C8—H8	116 (2)
C3—C2—H2	118 (2)	Cl1—C8—H8	108 (2)
C1—C2—H2	121 (2)	Cl2—C8—H8	103 (2)
C2—C3—C4	119.2 (3)	C3—C9—H9A	109.5
C2—C3—C9	119.6 (3)	C3—C9—H9B	109.5
C4—C3—C9	121.2 (3)	H9A—C9—H9B	109.5
C5—C4—C3	118.2 (3)	C3—C9—H9C	109.5
C5—C4—C10	120.4 (3)	H9A—C9—H9C	109.5
C3—C4—C10	121.4 (3)	H9B—C9—H9C	109.5
C6—C5—C4	123.2 (3)	C4—C10—H10A	109.5
C6—C5—H5	117 (3)	C4—C10—H10B	109.5
C4—C5—H5	119 (3)	H10A—C10—H10B	109.5
C5—C6—C1	118.2 (3)	C4—C10—H10C	109.5
C5—C6—H6	120 (2)	H10A—C10—H10C	109.5

C1—C6—H6	122 (2)	H10B—C10—H10C	109.5
C7—N1—C1—C6	31.6 (5)	C10—C4—C5—C6	177.8 (3)
C7—N1—C1—C2	−149.1 (3)	C4—C5—C6—C1	0.4 (5)
C6—C1—C2—C3	−1.8 (5)	C2—C1—C6—C5	1.6 (5)
N1—C1—C2—C3	178.8 (3)	N1—C1—C6—C5	−179.1 (3)
C1—C2—C3—C4	0.0 (5)	C1—N1—C7—O1	−4.0 (5)
C1—C2—C3—C9	−178.6 (3)	C1—N1—C7—C8	176.7 (3)
C2—C3—C4—C5	2.0 (5)	O1—C7—C8—Cl1	50.4 (4)
C9—C3—C4—C5	−179.4 (3)	N1—C7—C8—Cl1	−130.4 (3)
C2—C3—C4—C10	−178.1 (3)	O1—C7—C8—Cl2	−71.0 (3)
C9—C3—C4—C10	0.5 (5)	N1—C7—C8—Cl2	108.2 (3)
C3—C4—C5—C6	−2.2 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···O1^{i}	0.84 (4)	2.07 (4)	2.894 (3)	166 (3)

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

Fig. 1

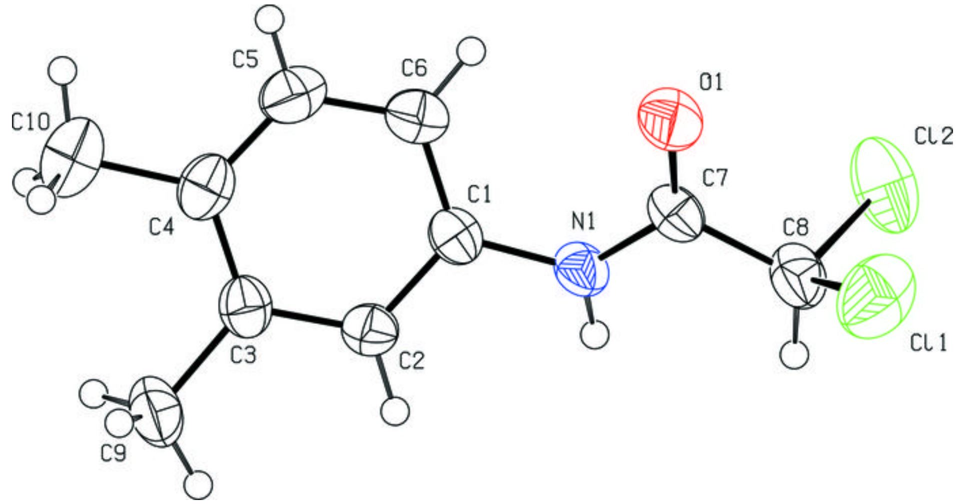

Fig. 2