organic compounds

N-(2-Chlorophenyl)-2,2,2-trimethyl-acetamide

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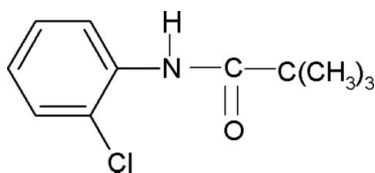Correspondence e-mail: gowdabt@yahoo.com

The conformation of the N—H bond in the structure of the title compound, $C_{11}H_{14}ClNO$, is *syn* to the *ortho*-chloro substituent, similar to that in 2,2,2-trimethyl-*N*-(2-methyl-phenyl)acetamide and the side-chain unsubstituted *N*-(2-chlorophenyl)acetamide, but in contrast with the *anti* conformation observed for *N*-(3-chlorophenyl)-2,2,2-trimethylacetamide. The bond parameters are also similar to those observed in related compounds and other acetanilides. The molecules are linked into chains through N—H⋯O hydrogen bonding.

Related literature

For related literature, see: Gowda *et al.* (2003, 2007, 2007*a*, 2007*b*).

Experimental

Crystal data

$C_{11}H_{14}ClNO$	V = 1152.6 (2) Å³
M_r = 211.68	Z = 4
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation
a = 10.159 (1) Å	μ = 0.30 mm⁻¹
b = 10.248 (1) Å	T = 302 (2) K
c = 11.071 (1) Å	$0.60 \times 0.16 \times 0.14$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	Reid, 1995)
	T_{min} = 0.840, T_{max} = 0.959
Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2006) using a multi-faceted crystal model (Clark &	6499 measured reflections
	2261 independent reflections
	1279 reflections with $I > 2\sigma(I)$
	R_{int} = 0.049

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.136	
S = 1.05	
2261 reflections	$\Delta\rho_{max}$ = 0.37 e Å⁻³
133 parameters	$\Delta\rho_{min}$ = −0.24 e Å⁻³
1 restraint	Absolute structure: Flack (1983), with 892 Friedel pairs
	Flack parameter: −0.07 (13)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O1ⁱ	0.841 (10)	2.23 (2)	2.973 (3)	148 (3)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2190).

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supplementary materials

N-(2-Chlorophenyl)-2,2,2-trimethylacetamide

B. T. Gowda, I. Svoboda and H. Fuess

Comment

The structure of *N*-(2-chlorophenyl)-2,2,2-trimethylacetamide (2CPTMA) has been determined as part of a study on the systematization of the crystal structures of N-aromatic amides (Gowda *et al.*, 2007, 2007*a*,*b*). The conformation of the N—H bond in 2CPTMA is *syn* to the *ortho*-chloro substituent (Fig. 1), similar to that in *N*-(2-methylphenyl)-2,2,2-trimethylacet-amide (2MPTMA) (Gowda *et al.*, 2007*a*) and the side chain unsubstituted *N*-(2-chlorophenyl)-acetamide (2CPA), but in contrast to the anti conformation observed for the *N*-(3-chlorophenyl)-2,2,2-trimethylacetamide (3CPTMA) (Gowda *et al.*, 2007). The geometric parameters of 2CPTMA are also similar to those of 2MPTMA, 2CPA, 3CPTMA and other acetanilides (Gowda *et al.*, 2007, 2007*a*,*b*). The molecules in 2CPTMA are packed into chains in the direction of *c* axis through N—H⋯O hydrogen bonds (Fig. 2 & Table 1).

Experimental

The title compound was prepared according to the literature method (Gowda *et al.*, 2003). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra (Gowda *et al.*, 2003). Single crystals of the title compound were obtained from an ethanolic solution and used for X-ray diffraction studies at room temperature.

Refinement

All H atoms attached to C atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (Caromatic) and 0.96 Å (Cmethyl) with $U_{iso}(H) = 1.2U_{eq}$(Caromatic) or $U_{iso}(H) = 1.5U_{eq}$(Cmethyl). H atom attached to N atom was located in difference Fourier maps and included in the subsequent refinement using restraint (N—H= 0.85 (1) Å) with $U_{iso}(H) = 1.2U_{eq}$(N).

Figures
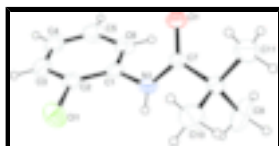
Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

Fig. 2. Partial packing view showing the formation of the chain through N—H⋯O Hydrogen bonding. H atoms not involved in hydrogen bondings have been omitted for clarity. Hydrogen bonds are shown as dashed lines. [Symmetry code: (i) *x* − 1/2, 1/2 − *y*, 2 − *z*].

N-(2-Chlorophenyl)-2,2,2-trimethylacetamide

Crystal data

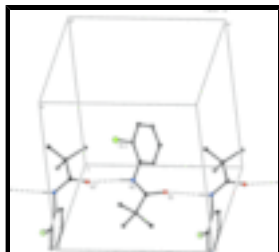|	
$C_{11}H_{14}ClNO$	$F_{000} = 448$
$M_r = 211.68$	$D_x = 1.220$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2ac 2ab	Cell parameters from 1749 reflections
$a = 10.159$ (1) Å	$\theta = 2.7$–20.5º
$b = 10.248$ (1) Å	$\mu = 0.30$ mm^{-1}
$c = 11.071$ (1) Å	$T = 302$ (2) K
$V = 1152.6$ (2) Å^3	Rod shape, colourless
$Z = 4$	$0.60 \times 0.16 \times 0.14$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	2261 independent reflections
Radiation source: Enhance (Mo) X-ray Source	1279 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.049$
Detector resolution: 8.4012 pixels mm^{-1}	$\theta_{max} = 26.4$º
$T = 302(2)$ K	$\theta_{min} = 2.7$º
Rotation method data acquisition using ω and φ scans	$h = -12\rightarrow9$
Absorption correction: analytical (CrysAlis RED; Oxford Diffraction, 2006) using a multifaceted crystal model (Clark & Reid, 1995)	$k = -11\rightarrow12$
$T_{min} = 0.840$, $T_{max} = 0.959$	$l = -10\rightarrow13$
6499 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.049$	$w = 1/[\sigma^2(F_o^2) + (0.0688P)^2 + 0.003P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.136$	$(\Delta/\sigma)_{max} = 0.002$
$S = 1.05$	$\Delta\rho_{max} = 0.37$ e Å^{-3}
2261 reflections	$\Delta\rho_{min} = -0.24$ e Å^{-3}
133 parameters	Extinction correction: none
1 restraint	Absolute structure: Flack (1983), with 892 Friedel pairs
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.07 (13)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	0.3757 (3)	0.1032 (3)	0.9165 (3)	0.0485 (9)
C2	0.3373 (3)	0.0764 (3)	0.7973 (3)	0.0529 (9)
C3	0.3577 (4)	−0.0442 (4)	0.7466 (4)	0.0701 (11)
H3	0.3290	−0.0615	0.6684	0.084*
C4	0.4203 (4)	−0.1384 (4)	0.8118 (4)	0.0790 (12)
H4	0.4354	−0.2197	0.7770	0.095*
C5	0.4610 (4)	−0.1155 (4)	0.9273 (5)	0.0776 (13)
H5	0.5042	−0.1805	0.9705	0.093*
C6	0.4375 (4)	0.0066 (3)	0.9809 (4)	0.0602 (10)
H6	0.4638	0.0219	1.0601	0.072*
C7	0.4429 (3)	0.3056 (3)	1.0162 (3)	0.0451 (7)
C8	0.3943 (3)	0.4347 (3)	1.0682 (3)	0.0533 (9)
C9	0.3016 (6)	0.4088 (4)	1.1719 (3)	0.1125 (19)
H9A	0.3464	0.3596	1.2331	0.169*
H9B	0.2271	0.3602	1.1434	0.169*
H9C	0.2723	0.4903	1.2052	0.169*
C10	0.3203 (5)	0.5103 (4)	0.9698 (4)	0.0949 (15)
H10A	0.2989	0.5959	0.9990	0.142*
H10B	0.2407	0.4647	0.9494	0.142*
H10C	0.3749	0.5176	0.8993	0.142*
C11	0.5068 (4)	0.5190 (4)	1.1055 (5)	0.0958 (16)
H11A	0.4737	0.6001	1.1367	0.144*
H11B	0.5620	0.5359	1.0369	0.144*
H11C	0.5570	0.4754	1.1669	0.144*
N1	0.3506 (3)	0.2264 (3)	0.9685 (3)	0.0541 (7)
H1	0.2739 (15)	0.253 (3)	0.954 (3)	0.065*
O1	0.5584 (2)	0.2731 (2)	1.0171 (3)	0.0663 (7)
Cl1	0.26026 (11)	0.19639 (10)	0.71284 (8)	0.0803 (4)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

C1	0.036 (2)	0.0512 (19)	0.058 (2)	−0.0058 (16)	0.0068 (18)	−0.0057 (16)
C2	0.0393 (19)	0.059 (2)	0.061 (2)	−0.0091 (16)	0.0096 (18)	−0.0019 (18)
C3	0.059 (2)	0.075 (3)	0.077 (3)	−0.008 (2)	0.008 (2)	−0.022 (2)
C4	0.069 (3)	0.063 (3)	0.105 (4)	−0.003 (2)	0.016 (3)	−0.031 (2)
C5	0.067 (3)	0.050 (2)	0.116 (4)	0.0075 (19)	0.001 (3)	0.004 (2)
C6	0.053 (2)	0.058 (2)	0.071 (2)	0.0044 (17)	0.000 (2)	0.002 (2)
C7	0.0353 (18)	0.0538 (18)	0.0463 (17)	−0.0028 (18)	0.0013 (16)	−0.0029 (18)
C8	0.047 (2)	0.0498 (19)	0.063 (2)	−0.0012 (17)	−0.0005 (18)	−0.0107 (17)
C9	0.176 (5)	0.088 (3)	0.073 (3)	−0.005 (3)	0.060 (3)	−0.015 (2)
C10	0.105 (3)	0.066 (2)	0.114 (3)	0.022 (2)	−0.029 (3)	−0.008 (3)
C11	0.071 (3)	0.074 (3)	0.143 (4)	0.004 (2)	−0.022 (3)	−0.034 (3)
N1	0.0377 (15)	0.0533 (16)	0.0713 (17)	0.0076 (14)	−0.0047 (16)	−0.0130 (15)
O1	0.0377 (13)	0.0658 (15)	0.0955 (17)	0.0029 (11)	−0.0005 (14)	−0.0217 (14)
Cl1	0.0859 (7)	0.0839 (7)	0.0712 (6)	−0.0095 (6)	−0.0137 (6)	0.0169 (5)

Geometric parameters (Å, °)

C1—C6	1.372 (5)	C7—C8	1.526 (5)
C1—C2	1.404 (5)	C8—C11	1.491 (5)
C1—N1	1.410 (4)	C8—C9	1.508 (5)
C2—C3	1.374 (5)	C8—C10	1.534 (5)
C2—Cl1	1.732 (3)	C9—H9A	0.9600
C3—C4	1.363 (6)	C9—H9B	0.9600
C3—H3	0.9300	C9—H9C	0.9600
C4—C5	1.365 (6)	C10—H10A	0.9600
C4—H4	0.9300	C10—H10B	0.9600
C5—C6	1.405 (5)	C10—H10C	0.9600
C5—H5	0.9300	C11—H11A	0.9600
C6—H6	0.9300	C11—H11B	0.9600
C7—O1	1.219 (4)	C11—H11C	0.9600
C7—N1	1.348 (4)	N1—H1	0.841 (10)
C6—C1—C2	118.3 (3)	C11—C8—C10	106.2 (3)
C6—C1—N1	121.1 (3)	C9—C8—C10	108.9 (3)
C2—C1—N1	120.6 (3)	C7—C8—C10	109.2 (3)
C3—C2—C1	121.2 (3)	C8—C9—H9A	109.5
C3—C2—Cl1	119.1 (3)	C8—C9—H9B	109.5
C1—C2—Cl1	119.6 (2)	H9A—C9—H9B	109.5
C4—C3—C2	119.4 (4)	C8—C9—H9C	109.5
C4—C3—H3	120.3	H9A—C9—H9C	109.5
C2—C3—H3	120.3	H9B—C9—H9C	109.5
C3—C4—C5	121.0 (4)	C8—C10—H10A	109.5
C3—C4—H4	119.5	C8—C10—H10B	109.5
C5—C4—H4	119.5	H10A—C10—H10B	109.5
C4—C5—C6	119.9 (4)	C8—C10—H10C	109.5
C4—C5—H5	120.1	H10A—C10—H10C	109.5
C6—C5—H5	120.1	H10B—C10—H10C	109.5
C1—C6—C5	120.1 (4)	C8—C11—H11A	109.5
C1—C6—H6	120.0	C8—C11—H11B	109.5
C5—C6—H6	120.0	H11A—C11—H11B	109.5

O1—C7—N1	120.5 (3)	C8—C11—H11C	109.5
O1—C7—C8	123.1 (3)	H11A—C11—H11C	109.5
N1—C7—C8	116.4 (3)	H11B—C11—H11C	109.5
C11—C8—C9	111.7 (4)	C7—N1—C1	125.0 (3)
C11—C8—C7	111.0 (3)	C7—N1—H1	122 (2)
C9—C8—C7	109.7 (3)	C1—N1—H1	112 (2)

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1[i]	0.841 (10)	2.23 (2)	2.973 (3)	148 (3)

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

Fig. 1

Fig. 2