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N-(2,3-Dichlorophenyl)-2,2,2-trimethylacetamide

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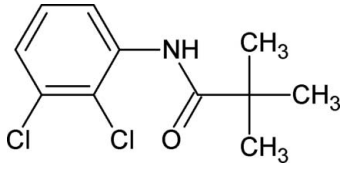
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.005 Å; R factor = 0.061; wR factor = 0.176; data-to-parameter ratio = 15.9.

The conformation of the N—H bond in the title compound (23DCPTMA), $C_{11}H_{13}Cl_2NO$, is *syn* to both the 2-chloro and the 3-chloro groups, similar to that in *N*-(2,3-dichlorophenyl)-acetamide (23DCPA), but in contrast to the *anti* conformation with respect to the 3-chloro substituent in *N*-(3-chlorophenyl)-2,2,2-trimethylacetamide (3CPTMA). The bond parameters of 23DCPTMA are similar to those in 3CPTMA, 23DCPA and other acetanilides. The molecules in 23DCPTMA are linked into chains through N—H···O hydrogen bonding.

Related literature

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



Experimental

Crystal data

$C_{11}H_{13}Cl_2NO$
M_r = 246.12
Monoclinic, $P2_1/c$
a = 11.206 (3) Å
b = 11.139 (3) Å
c = 10.075 (2) Å
β = 102.70 (3)°
V = 1226.8 (5) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 4.55 mm^{-1}
T = 299 (2) K
0.60 × 0.30 × 0.20 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.264, T_{max} = 0.402
2957 measured reflections
2174 independent reflections
1680 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.176
S = 1.06
2174 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.50 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N7—H7N···O6^{i}	0.86	2.13	2.972 (3)	165

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

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC Software*; data reduction: *REDU4* (Stoe & Cie, 1987); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); 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: WN2185).

References

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supplementary materials

Acta Cryst. (2007). E**63**, o3788 [**doi:10.1107/S160053680703930X**]

N-(2,3-Dichlorophenyl)-2,2,2-trimethylacetamide

B. T. Gowda, S. Foro and H. Fuess

Comment

As part of a study of the effect of ring and side chain substitutions on the solid state structures of aromatic amides (Gowda *et al.*, 2007*a*,b), the crystal structure of *N*-(2,3-dichlorophenyl)-2,2,2-trimethylacetamide (23DCPTMA) has been determined. The conformation of the N—H bond in 23DCPTMA is *syn* to both the 2-chloro and 3-chloro groups (Fig. 1), similar to that in *N*-(2,3-dichlorophenyl)-acetamide (23DCPA)(Gowda *et al.*, 2007*b*), but in contrast to the *anti* conformation with respect to the 3-chloro substituent in *N*-(3-chlorophenyl)- 2,2,2-trimethylacetamide (3CPTMA) (Gowda *et al.*, 2007*a*). Intermolecular N—H···O hydrogen bonds (Table 1) link the molecules into chains (Fig. 2).

Experimental

The title compound was prepared according to a literature method (Shilpa & Gowda, 2007). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared and NMR spectra (Shilpa & Gowda, 2007). Single crystals of the title compound were obtained by slow evaporation of an ethanolic solution (2 g in about 30 ml e thanol) and used for the X-ray diffraction study at room temperature.

Refinement

All H atoms were positioned geometrically and refined as riding with C—H = 0.93–0.96 Å and N—H = 0.86 Å; U_{iso}(H) = 1.2U_{eq}(C,*N*).

Figures

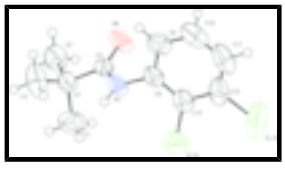

Fig. 1. The molecular structure of the title compound, showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radius.

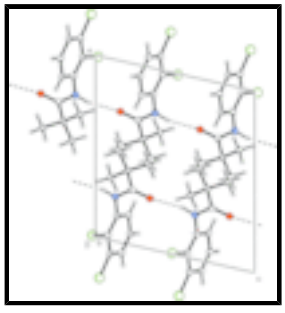

Fig. 2. Molecular packing of the title compound, with hydrogen bonding shown as dashed lines.

N-(2,3-Dichlorophenyl)-2,2,2-trimethylacetamide

Crystal data

$C_{11}H_{13}Cl_2NO$	F_{000} = 512
M_r = 246.12	D_x = 1.333 Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation λ = 1.54180 Å
Hall symbol: -P 2ybc	Cell parameters from 24 reflections
a = 11.206 (3) Å	θ = 5.7–19.5º
b = 11.139 (3) Å	μ = 4.55 mm^{-1}
c = 10.075 (2) Å	T = 299 (2) K
β = 102.70 (3)º	Rod, colourless
V = 1226.8 (5) Å^3	0.60 × 0.30 × 0.20 mm
Z = 4	

Data collection

Enraf–Nonius CAD-4 diffractometer	R_{int} = 0.029
Radiation source: fine-focus sealed tube	θ_{max} = 66.9º
Monochromator: graphite	θ_{min} = 4.0º
T = 299(2) K	h = −13→13
ω/2θ scans	k = −3→13
Absorption correction: ψ scan (North *et al.*, 1968)	l = 0→12
T_{min} = 0.264, T_{max} = 0.402	3 standard reflections
2957 measured reflections	every 120 min
2174 independent reflections	intensity decay: 1.0%
1680 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.061	$w = 1/[\sigma^2(F_o^2) + (0.1081P)^2 + 0.276P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.176	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.06	$\Delta\rho_{max}$ = 0.30 e Å^{-3}
2174 reflections	$\Delta\rho_{min}$ = −0.49 e Å^{-3}
137 parameters	Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0058 (11)
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}
Cl14	1.22112 (9)	0.32462 (14)	0.47441 (15)	0.1266 (6)
Cl15	0.99510 (9)	0.17412 (8)	0.51766 (9)	0.0841 (4)
O6	0.7204 (2)	0.2107 (2)	0.15772 (18)	0.0698 (6)
N7	0.7557 (2)	0.2709 (2)	0.3746 (2)	0.0570 (6)
H7N	0.7322	0.2703	0.4503	0.068*
C1	0.6547 (4)	0.0286 (4)	0.4031 (4)	0.1053 (14)
H1A	0.5933	−0.0254	0.4209	0.126*
H1B	0.6931	0.0687	0.4858	0.126*
H1C	0.7151	−0.0159	0.3691	0.126*
C2	0.5355 (3)	0.0561 (3)	0.1681 (3)	0.0802 (10)
H2A	0.5961	0.0112	0.1351	0.096*
H2B	0.4982	0.1136	0.1007	0.096*
H2C	0.4741	0.0023	0.1864	0.096*
C3	0.5000 (4)	0.1915 (4)	0.3513 (5)	0.0960 (13)
H3A	0.4383	0.1376	0.3686	0.115*
H3B	0.4630	0.2499	0.2848	0.115*
H3C	0.5382	0.2315	0.4341	0.115*
C4	0.5956 (2)	0.1210 (2)	0.2978 (3)	0.0525 (6)
C5	0.6947 (2)	0.2046 (2)	0.2697 (2)	0.0466 (6)
C8	0.8579 (2)	0.3419 (2)	0.3625 (2)	0.0537 (7)
C9	0.8410 (3)	0.4482 (3)	0.2922 (3)	0.0674 (8)
H9	0.7623	0.4743	0.2531	0.081*
C10	0.9402 (4)	0.5161 (3)	0.2796 (3)	0.0816 (10)
H10	0.9282	0.5883	0.2322	0.098*
C11	1.0561 (4)	0.4787 (3)	0.3358 (4)	0.0854 (11)
H11	1.1226	0.5252	0.3264	0.102*
C12	1.0746 (3)	0.3726 (3)	0.4063 (3)	0.0735 (9)
C13	0.9751 (3)	0.3034 (3)	0.4223 (3)	0.0586 (7)

Atomic displacement parameters (Å^2)

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

Cl14	0.0569 (5)	0.1743 (13)	0.1510 (12)	−0.0080 (6)	0.0279 (6)	−0.0386 (9)
Cl15	0.0899 (6)	0.0854 (6)	0.0783 (6)	0.0121 (4)	0.0208 (4)	0.0081 (4)
O6	0.0851 (14)	0.0934 (15)	0.0356 (9)	−0.0323 (12)	0.0234 (9)	−0.0110 (9)
N7	0.0609 (12)	0.0793 (15)	0.0333 (10)	−0.0228 (11)	0.0158 (9)	−0.0086 (9)
C1	0.109 (3)	0.097 (3)	0.098 (3)	−0.031 (2)	0.000 (2)	0.045 (2)
C2	0.079 (2)	0.094 (2)	0.0668 (19)	−0.0310 (18)	0.0145 (16)	−0.0173 (17)
C3	0.078 (2)	0.095 (3)	0.131 (3)	−0.0221 (19)	0.059 (2)	−0.034 (2)
C4	0.0542 (13)	0.0578 (15)	0.0476 (13)	−0.0097 (11)	0.0155 (11)	−0.0023 (11)
C5	0.0488 (12)	0.0558 (14)	0.0361 (12)	−0.0020 (10)	0.0114 (9)	−0.0021 (9)
C8	0.0606 (15)	0.0651 (16)	0.0375 (12)	−0.0170 (12)	0.0157 (11)	−0.0112 (11)
C9	0.082 (2)	0.0704 (18)	0.0499 (14)	−0.0113 (15)	0.0140 (13)	−0.0026 (13)
C10	0.117 (3)	0.071 (2)	0.0636 (18)	−0.029 (2)	0.0357 (19)	−0.0050 (15)
C11	0.095 (3)	0.091 (3)	0.084 (2)	−0.046 (2)	0.049 (2)	−0.0300 (19)
C12	0.0599 (16)	0.096 (2)	0.0704 (19)	−0.0200 (16)	0.0268 (14)	−0.0292 (17)
C13	0.0625 (16)	0.0688 (17)	0.0480 (14)	−0.0087 (12)	0.0201 (12)	−0.0150 (12)

Geometric parameters (Å, °)

Cl14—C12	1.720 (4)	C3—C4	1.519 (4)
Cl15—C13	1.719 (3)	C3—H3A	0.9600
O6—C5	1.226 (3)	C3—H3B	0.9600
N7—C5	1.346 (3)	C3—H3C	0.9600
N7—C8	1.419 (3)	C4—C5	1.523 (3)
N7—H7N	0.8600	C8—C9	1.371 (4)
C1—C4	1.522 (4)	C8—C13	1.387 (4)
C1—H1A	0.9600	C9—C10	1.374 (5)
C1—H1B	0.9600	C9—H9	0.9300
C1—H1C	0.9600	C10—C11	1.362 (6)
C2—C4	1.516 (4)	C10—H10	0.9300
C2—H2A	0.9600	C11—C12	1.371 (5)
C2—H2B	0.9600	C11—H11	0.9300
C2—H2C	0.9600	C12—C13	1.393 (4)
C5—N7—C8	121.2 (2)	C2—C4—C5	109.6 (2)
C5—N7—H7N	119.4	C3—C4—C5	110.5 (2)
C8—N7—H7N	119.4	C1—C4—C5	108.7 (2)
C4—C1—H1A	109.5	O6—C5—N7	120.6 (2)
C4—C1—H1B	109.5	O6—C5—C4	122.3 (2)
H1A—C1—H1B	109.5	N7—C5—C4	117.0 (2)
C4—C1—H1C	109.5	C9—C8—C13	120.1 (3)
H1A—C1—H1C	109.5	C9—C8—N7	120.2 (3)
H1B—C1—H1C	109.5	C13—C8—N7	119.7 (2)
C4—C2—H2A	109.5	C8—C9—C10	120.0 (3)
C4—C2—H2B	109.5	C8—C9—H9	120.0
H2A—C2—H2B	109.5	C10—C9—H9	120.0
C4—C2—H2C	109.5	C11—C10—C9	120.7 (4)
H2A—C2—H2C	109.5	C11—C10—H10	119.7
H2B—C2—H2C	109.5	C9—C10—H10	119.7
C4—C3—H3A	109.5	C10—C11—C12	120.0 (3)
C4—C3—H3B	109.5	C10—C11—H11	120.0

H3A—C3—H3B	109.5	C12—C11—H11	120.0
C4—C3—H3C	109.5	C11—C12—C13	120.2 (3)
H3A—C3—H3C	109.5	C11—C12—Cl14	119.8 (3)
H3B—C3—H3C	109.5	C13—C12—Cl14	120.0 (3)
C2—C4—C3	109.8 (3)	C8—C13—C12	119.0 (3)
C2—C4—C1	108.7 (3)	C8—C13—Cl15	119.8 (2)
C3—C4—C1	109.6 (3)	C12—C13—Cl15	121.3 (3)
C8—N7—C5—O6	−5.4 (4)	C8—C9—C10—C11	0.3 (5)
C8—N7—C5—C4	173.3 (2)	C9—C10—C11—C12	−0.3 (5)
C2—C4—C5—O6	−2.5 (4)	C10—C11—C12—C13	−0.8 (5)
C3—C4—C5—O6	−123.5 (3)	C10—C11—C12—Cl14	178.8 (3)
C1—C4—C5—O6	116.2 (3)	C9—C8—C13—C12	−1.8 (4)
C2—C4—C5—N7	178.8 (3)	N7—C8—C13—C12	178.3 (2)
C3—C4—C5—N7	57.8 (4)	C9—C8—C13—Cl15	176.6 (2)
C1—C4—C5—N7	−62.5 (4)	N7—C8—C13—Cl15	−3.3 (3)
C5—N7—C8—C9	75.9 (4)	C11—C12—C13—C8	1.9 (4)
C5—N7—C8—C13	−104.3 (3)	Cl14—C12—C13—C8	−177.8 (2)
C13—C8—C9—C10	0.8 (4)	C11—C12—C13—Cl15	−176.5 (2)
N7—C8—C9—C10	−179.4 (3)	Cl14—C12—C13—Cl15	3.8 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7—H7N···O6^{i}	0.86	2.13	2.972 (3)	165

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

Fig. 1

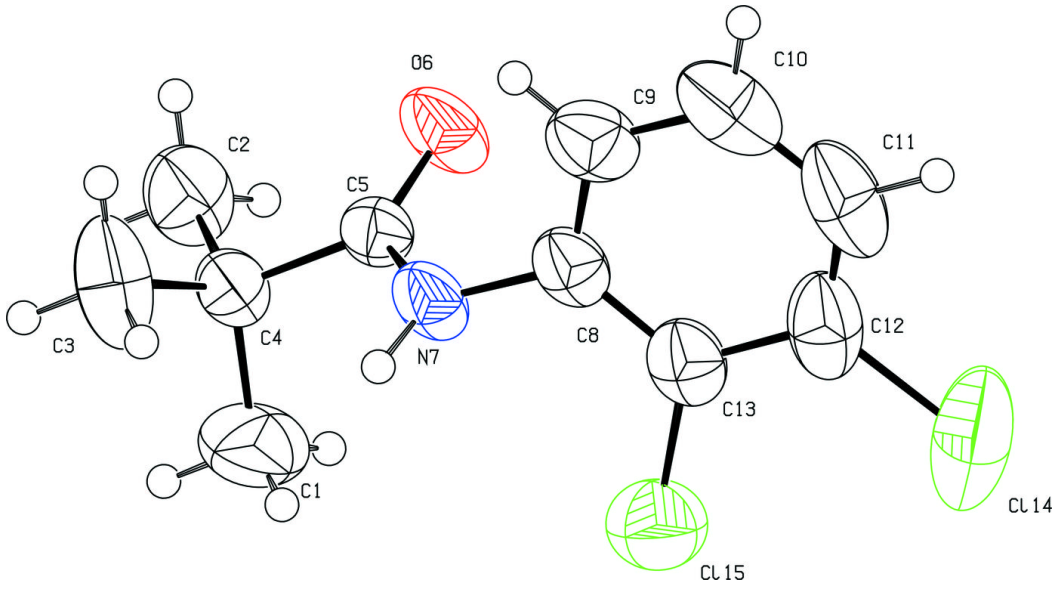

Fig. 2