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2,2-Dimethyl-*N*-(2,4,6-trichlorophenyl)-acetamide

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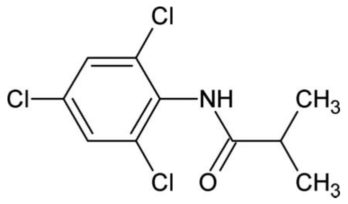
Key indicators: single-crystal X-ray study; *T* = 297 K; mean σ(C–C) = 0.005 Å; *R* factor = 0.060; *wR* factor = 0.126; data-to-parameter ratio = 17.0.

The structure of the title compound, $C_{10}H_{10}Cl_3NO$, is similar to that of *N*-(2,4,6-trichlorophenyl)-chloro/methyl-acetamides and other acetanilides, with somewhat different geometric parameters. Comparison of the geometric parameters of the title compound with those of the ring and side-chain-substituted acetanilides reveals that substitution either in the benzene ring or in the side chain of the amides changes some of the space groups but also some of the bond lengths, and bond and torsion angles. The molecules in the title compound are packed into chains through N—H···O hydrogen bonding.

Related literature

For related literature, see: Gowda *et al.* (2000, 2006); Gowda, Svoboda & Fuess (2004); Gowda, Usha & Jyothi (2004); Gowda, Foro *et al.* (2007); Gowda, Kozisek *et al.* (2007); Mahalakshmi *et al.* (2002); Nyburg *et al.* (1987).



Experimental

Crystal data

$C_{10}H_{10}Cl_3NO$
M_r = 266.54
Monoclinic, $P2_1/n$
a = 4.7310 (6) Å
b = 24.042 (2) Å
c = 10.799 (1) Å
β = 98.01 (1)°
V = 1216.3 (2) Å^3
Z = 4
Mo *K*α radiation
μ = 0.73 mm^{-1}
T = 297 (2) K
0.50 × 0.12 × 0.06 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector
Absorption correction: multi-scan [*CrysAlis RED* (Oxford Diffraction, 2006); analytical numeric absorption correction using a multifaceted crystal model based on expressions derived by Clark & Reid (1995)]
T_{min} = 0.713, T_{max} = 0.958
8334 measured reflections
2460 independent reflections
1574 reflections with $I > 2\sigma(I)$
R_{int} = 0.039

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.126
S = 1.08
2460 reflections
145 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.79 (3)	2.12 (3)	2.889 (3)	165 (3)

Symmetry code: (i) *x* − 1, *y*, *z*.

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*.

BTG thanks the Alexander von Humboldt Foundation, Bonn, Germany for extensions of his research fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2020).

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supplementary materials

Acta Cryst. (2007). E**63**, o3366   [**doi:10.1107/S1600536807031728**]

2,2-Dimethyl-*N*-(2,4,6-trichlorophenyl)acetamide

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

Comment

As part of a study on the effect of side chain and ring substitutions on the solid state structures of a chemically and biologically significant class of compounds (Gowda *et al.*, 2000; 2004*b*; 2006; 2007*a*, *b*), in the present work, the crystal structure of *N*-(2,4,6-trichlorophenyl)-2,2-dimethylacetamide (246TCPDMA) has been determined. The structure of 246TCPDMA (Fig. 1) is similar to those of *N*-(2,4,6-trichlorophenyl)-chloro/methyl-acetamides and other acetanilides, with somewhat different geometric parameters (Gowda *et al.*, 2000; 2004*b*; 2006; 2007*a*, *b*; Mahalakshmi *et al.*, 2002; Nyburg *et al.*, 1987). The comparison of the geometric parameters of the title compound with those of the ring and side chain substituted acetanilides revealed that substitution either in the benzene ring or in the side chain of the amides not only changes the space groups but also some of the bond lengths as well as bond and torsional angles (Gowda *et al.*, 2000; 2004*a*; 2006; 2007*a*, *b*). But to draw general conclusions further substantive data are to be collected with varying substitution patterns. The molecules in the title compound are packed into chains through N—H···O hydrogen bonding (Table 1 & Fig.2).

Experimental

The title compound was prepared according to the literature method (Gowda *et al.*, 2004*b*). The purity of the compound was checked by determining its melting point. The compound was further characterized by recording its infrared and NMR spectra (Gowda *et al.*, 2004*b*). Single crystals of the title compound were obtained from a slow evaporation of an ethanolic solution (2 g in about 30 ml e thanol) and used for X-ray diffraction studies at room temperature.

Refinement

The H atoms were located from the difference Fourier map and their positions refined with $U_{iso}(H) = 1.2\ U_{eq}$ (parent atom).

Figures

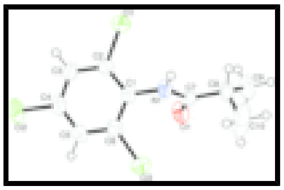

Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

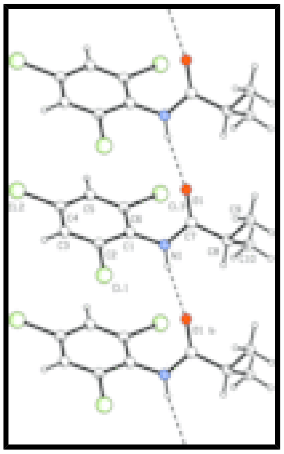

Fig. 2. Hydrogen bonding in the title compound. Hydrogen bonds are shown as dashed lines.

2,2-Dimethyl-*N*-(2,4,6-trichlorophenyl)acetamide

Crystal data

$C_{10}H_{10}Cl_3NO$

$M_r = 266.54$

Monoclinic, $P2_1/n$

Hall symbol: -P2yn

$a = 4.7310$ (6) Å

$b = 24.042$ (2) Å

$c = 10.799$ (1) Å

$\beta = 98.01$ (1)°

$V = 1216.3$ (2) Å^3

$Z = 4$

$F_{000} = 544$

$D_x = 1.456$ Mg m^{-3}

Mo *K*α radiation
$\lambda = 0.71073$ Å

Cell parameters from 2291 reflections

$\theta = 2.6$–24.4°

$\mu = 0.73$ mm^{-1}

$T = 297$ (2) K

Needle, colourless

0.50 × 0.12 × 0.06 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector

Radiation source: Enhance (Mo) X-ray Source

Monochromator: graphite

Detector resolution: 8.4012 pixels mm^{-1}

$T = 297(2)$ K

Rotation method data acquisition using ω and φ scans

Absorption correction: multi-scan [CrysAlis RED (Oxford Diffraction, 2006); analytical numeric absorption correction using a multifaceted crystal model based on expressions derived by Clark & Reid (1995)]

$T_{min} = 0.713$, $T_{max} = 0.958$

8334 measured reflections

2460 independent reflections

1574 reflections with $I > 2\sigma(I)$

$R_{int} = 0.039$

$\theta_{max} = 26.4$°

$\theta_{min} = 4.2$°

$h = -5 \rightarrow 5$

$k = -29 \rightarrow 27$

$l = -13 \rightarrow 13$

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
$R[F^2 > 2\sigma(F^2)] = 0.060$	$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2 + 1.2703P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.126$	$(\Delta/\sigma)_{max} = 0.002$
$S = 1.08$	$\Delta\rho_{max} = 0.36$ e Å^{-3}
2460 reflections	$\Delta\rho_{min} = -0.34$ e Å^{-3}
145 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: 0.0137 (15)
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 $F^2 > 2sigma(F^2)$ 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	0.8638 (6)	0.09310 (12)	0.2959 (3)	0.0344 (7)
C2	0.7415 (7)	0.06534 (13)	0.1894 (3)	0.0387 (8)
C3	0.8267 (7)	0.01277 (13)	0.1585 (3)	0.0467 (9)
H3	0.7398	−0.0051	0.0868	0.056*
C4	1.0425 (8)	−0.01217 (13)	0.2364 (4)	0.0504 (9)
C5	1.1702 (8)	0.01276 (14)	0.3435 (3)	0.0516 (9)
H5	1.3160	−0.0050	0.3954	0.062*
C6	1.0776 (7)	0.06514 (14)	0.3730 (3)	0.0427 (8)
C7	0.9518 (7)	0.19206 (13)	0.3283 (3)	0.0380 (8)
C8	0.8228 (7)	0.24621 (14)	0.3627 (4)	0.0503 (9)
H8	0.624 (8)	0.2474 (14)	0.326 (3)	0.060*
C9	0.9660 (9)	0.29551 (15)	0.3124 (5)	0.0736 (13)
H9A	0.9525	0.2925	0.2231	0.088*
H9B	1.1633	0.2964	0.3484	0.088*
H9C	0.8736	0.3291	0.3335	0.088*

C10	0.8362 (14)	0.2489 (2)	0.5023 (5)	0.114 (2)
H10A	1.0320	0.2500	0.5403	0.137*
H10B	0.7454	0.2166	0.5311	0.137*
H10C	0.7396	0.2817	0.5247	0.137*
N1	0.7771 (5)	0.14716 (11)	0.3235 (3)	0.0378 (7)
H1	0.612 (7)	0.1525 (14)	0.323 (3)	0.045*
O1	1.1986 (5)	0.18779 (9)	0.3089 (3)	0.0553 (7)
Cl1	0.4698 (2)	0.09734 (4)	0.09091 (9)	0.0612 (3)
Cl2	1.1579 (3)	−0.07790 (4)	0.19711 (12)	0.0811 (4)
Cl3	1.2321 (2)	0.09475 (4)	0.51250 (9)	0.0664 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0292 (16)	0.0317 (16)	0.0439 (17)	−0.0019 (14)	0.0108 (14)	−0.0012 (15)
C2	0.0346 (17)	0.0366 (18)	0.0439 (18)	−0.0016 (14)	0.0025 (15)	0.0015 (15)
C3	0.051 (2)	0.0364 (18)	0.053 (2)	−0.0018 (17)	0.0069 (17)	−0.0129 (16)
C4	0.055 (2)	0.0306 (17)	0.067 (2)	0.0045 (17)	0.0132 (19)	−0.0036 (17)
C5	0.054 (2)	0.0374 (19)	0.060 (2)	0.0092 (17)	−0.0016 (18)	0.0010 (17)
C6	0.0432 (19)	0.0400 (19)	0.0445 (19)	−0.0037 (16)	0.0045 (16)	−0.0009 (15)
C7	0.0301 (17)	0.0353 (17)	0.0480 (19)	−0.0023 (15)	0.0033 (15)	−0.0055 (15)
C8	0.0336 (18)	0.0375 (19)	0.081 (3)	−0.0016 (16)	0.0139 (19)	−0.0164 (18)
C9	0.078 (3)	0.037 (2)	0.109 (4)	0.002 (2)	0.022 (3)	−0.002 (2)
C10	0.198 (6)	0.067 (3)	0.092 (4)	−0.006 (4)	0.069 (4)	−0.029 (3)
N1	0.0254 (13)	0.0336 (14)	0.0556 (17)	−0.0005 (12)	0.0095 (13)	−0.0090 (12)
O1	0.0287 (13)	0.0439 (14)	0.096 (2)	−0.0027 (11)	0.0178 (12)	−0.0062 (13)
Cl1	0.0599 (6)	0.0559 (6)	0.0614 (6)	0.0128 (5)	−0.0142 (5)	−0.0093 (5)
Cl2	0.0946 (9)	0.0417 (5)	0.1062 (9)	0.0224 (5)	0.0108 (7)	−0.0166 (6)
Cl3	0.0807 (8)	0.0583 (6)	0.0527 (6)	0.0012 (5)	−0.0165 (5)	−0.0049 (5)

Geometric parameters (Å, °)

C1—C2	1.385 (4)	C7—N1	1.356 (4)
C1—C6	1.391 (4)	C7—C8	1.506 (4)
C1—N1	1.407 (4)	C8—C10	1.501 (6)
C2—C3	1.382 (4)	C8—C9	1.504 (5)
C2—Cl1	1.731 (3)	C8—H8	0.97 (4)
C3—C4	1.368 (5)	C9—H9A	0.9600
C3—H3	0.9300	C9—H9B	0.9600
C4—C5	1.367 (5)	C9—H9C	0.9600
C4—Cl2	1.743 (3)	C10—H10A	0.9600
C5—C6	1.385 (5)	C10—H10B	0.9600
C5—H5	0.9300	C10—H10C	0.9600
C6—Cl3	1.733 (3)	N1—H1	0.79 (3)
C7—O1	1.219 (3)		
C2—C1—C6	116.7 (3)	C10—C8—C9	111.8 (4)
C2—C1—N1	121.4 (3)	C10—C8—C7	108.8 (4)
C6—C1—N1	121.9 (3)	C9—C8—C7	111.9 (3)

C3—C2—C1	122.6 (3)	C10—C8—H8	108 (2)
C3—C2—Cl1	118.4 (3)	C9—C8—H8	107 (2)
C1—C2—Cl1	119.0 (2)	C7—C8—H8	109 (2)
C4—C3—C2	118.1 (3)	C8—C9—H9A	109.5
C4—C3—H3	121.0	C8—C9—H9B	109.5
C2—C3—H3	121.0	H9A—C9—H9B	109.5
C5—C4—C3	122.2 (3)	C8—C9—H9C	109.5
C5—C4—Cl2	119.1 (3)	H9A—C9—H9C	109.5
C3—C4—Cl2	118.7 (3)	H9B—C9—H9C	109.5
C4—C5—C6	118.5 (3)	C8—C10—H10A	109.5
C4—C5—H5	120.8	C8—C10—H10B	109.5
C6—C5—H5	120.8	H10A—C10—H10B	109.5
C5—C6—C1	121.9 (3)	C8—C10—H10C	109.5
C5—C6—Cl3	117.7 (3)	H10A—C10—H10C	109.5
C1—C6—Cl3	120.4 (3)	H10B—C10—H10C	109.5
O1—C7—N1	121.3 (3)	C7—N1—C1	123.3 (3)
O1—C7—C8	123.1 (3)	C7—N1—H1	118 (3)
N1—C7—C8	115.6 (3)	C1—N1—H1	118 (3)

C6—C1—C2—C3	−0.8 (5)	C2—C1—C6—C5	1.8 (5)
N1—C1—C2—C3	178.5 (3)	N1—C1—C6—C5	−177.6 (3)
C6—C1—C2—Cl1	178.8 (2)	C2—C1—C6—Cl3	−176.8 (2)
N1—C1—C2—Cl1	−1.8 (4)	N1—C1—C6—Cl3	3.9 (4)
C1—C2—C3—C4	−0.6 (5)	O1—C7—C8—C10	−96.7 (4)
Cl1—C2—C3—C4	179.7 (3)	N1—C7—C8—C10	81.4 (4)
C2—C3—C4—C5	1.3 (5)	O1—C7—C8—C9	27.4 (5)
C2—C3—C4—Cl2	−178.7 (3)	N1—C7—C8—C9	−154.5 (3)
C3—C4—C5—C6	−0.4 (6)	O1—C7—N1—C1	−0.3 (5)
Cl2—C4—C5—C6	179.6 (3)	C8—C7—N1—C1	−178.4 (3)
C4—C5—C6—C1	−1.2 (5)	C2—C1—N1—C7	−115.8 (3)
C4—C5—C6—Cl3	177.4 (3)	C6—C1—N1—C7	63.5 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.79 (3)	2.12 (3)	2.889 (3)	165 (3)

Symmetry codes: (i) *x*−1, *y*, *z*.

Fig. 1

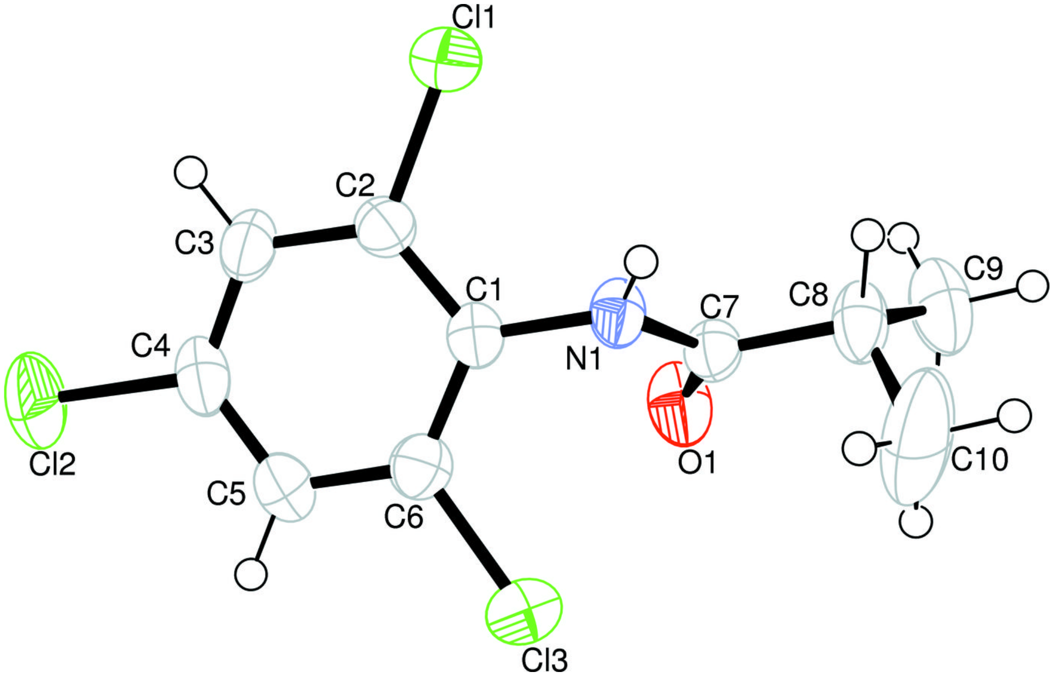

Fig. 2