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2,2,2-Trichloro-*N*-(2,4-dichlorophenyl)-acetamide

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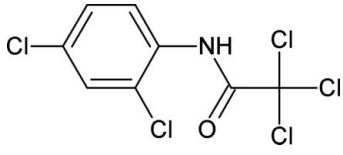
Key indicators: single-crystal X-ray study; T = 299 K; mean σ(C–C) = 0.005 Å; R factor = 0.055; wR factor = 0.156; data-to-parameter ratio = 14.7.

The conformation of the N—H bond in the structure of the title compound, $C_8H_4Cl_5NO$, is *syn* to the *ortho*-chloro substituent, similar to that observed in *N*-(2-chlorophenyl)-acetamide, *N*-(2,4-dichlorophenyl)acetamide, 2,2,2-trichloro-*N*-(2-chlorophenyl)acetamide and *N*-(2-chlorophenyl)-2,2,2-trimethylacetamide. The bond parameters are similar to those in other acetanilides. The amide H atom is involved in two intramolecular hydrogen bonds with the *ortho* ring Cl atom and one of the Cl atoms of the CCl_3 group.

Related literature

For related literature, see: Gowda *et al.* (2000); Gowda, Kozisek *et al.* (2007); Gowda *et al.* (2007*a*,*b*,*c*); Pies *et al.* (1971); Shilpa & Gowda (2007).



Experimental

Crystal data

$C_8H_4Cl_5NO$
M_r = 307.37
Orthorhombic, *Pbca*
a = 14.306 (2) Å
b = 8.625 (2) Å
c = 18.317 (4) Å
V = 2260.1 (8) Å^3
Z = 8
Cu $K\alpha$ radiation
μ = 11.47 mm^{-1}
T = 299 (2) K
0.40 × 0.25 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.091, T_{max} = 0.199
(expected range = 0.026–0.057)
3958 measured reflections
2017 independent reflections
1780 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
3 standard reflections
frequency: 120 min
intensity decay: 1.0%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.156
S = 1.04
2017 reflections
137 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.61 e Å^{-3}
$\Delta\rho_{min}$ = −0.57 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N7—H7N···Cl3	0.86	2.49	2.953 (3)	115
N7—H7N···Cl14	0.86	2.44	2.916 (3)	115

Data collection: *CAD-4-PC* (Enraf–Nonius, 1996); cell refinement: *CAD-4-PC*; 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: *ORTEP-3* (Farrugia, 1997) and *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: BT2420).

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supplementary materials

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2,2,2-Trichloro-*N*-(2,4-dichlorophenyl)acetamide

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Comment

In the present work, the structure of *N*-(2,4-dichlorophenyl)-2,2,2- trichloroacetamide has been determined as part of a study in the direction of systematization of the crystal structures of N-aromatic amides (Gowda *et al.*, 2000; 2007*a*, *b*, *c*, *d*). The conformation of the N—H bond is *syn* to the *ortho*-chloro substituent (Fig. 1), similar to that observed in *N*-(2-chlorophenyl)-acetamide (Gowda *et al.*, 2007*c*), *N*-(2,4-DiChlorophenyl)-acetamide (Gowda *et al.*, 2007*d*), *N*-(2-chlorophenyl)-2,2,2- trichloroacetamide (Gowda *et al.*, 2000) and *N*-(2-chlorophenyl)- 2,2,2-trimethylacetamide (Gowda *et al.*, 2007*b*). The geometric parameters are similar to those in other acetanilides (Gowda *et al.*, 2000; 2007*a*, *b*, *c*, *d*). The amide H is involved in two intra-molecular hydrogen bonding with the *ortho* ring Cl atom and one of the Cl atoms of the CCl_3 group (Fig. 1) (Fig. 2 & Table 1).

Experimental

The title compound was prepared according to the literature method (Shilpa & Gowda, 2007). The purity of the compound was checked by determining its melting point. It was characterized by recording its infrared, NMR (Shilpa & Gowda, 2007) and NQR spectra (Pies *et al.*, 1971). Single crystals of the title compound were obtained from a slow evaporation of its 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 positioned with idealized geometry using a riding model with N—H = 0.86 Å and C—H = 0.93 Å]. U_{iso}(H) values were set equal to 1.2 U_{eq} of the parent atom.

Figures

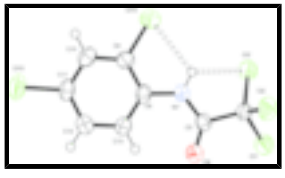

Fig. 1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

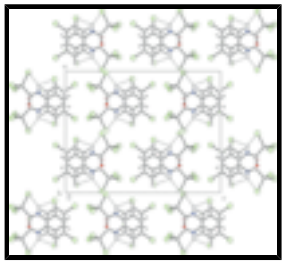

Fig. 2. Crystal packing of the title compound. Hydrogen bonds are shown as dashed lines.

2,2,2-Trichloro-*N*-(2,4-dichlorophenyl)acetamide

Crystal data

$C_8H_4Cl_5NO$	F_{000} = 1216
M_r = 307.37	D_x = 1.807 Mg m^{-3}
Orthorhombic, *Pbca*	Cu *K*α radiation λ = 1.54180 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 25 reflections
a = 14.306 (2) Å	θ = 5.7–19.1°
b = 8.625 (2) Å	μ = 11.47 mm^{-1}
c = 18.317 (4) Å	*T* = 299 (2) K
V = 2260.1 (8) Å^3	Rod, colourless
Z = 8	0.40 × 0.25 × 0.25 mm

Data collection

Enraf-Nonius CAD-4 diffractometer	R_{int} = 0.034
Radiation source: fine-focus sealed tube	θ_{max} = 67.0°
Monochromator: graphite	θ_{min} = 4.8°
T = 299(2) K	*h* = −17→0
ω/2θ scans	*k* = −10→0
Absorption correction: Psi-scan (North *et al.*, 1968)	*l* = −21→21
T_{min} = 0.091, T_{max} = 0.199	3 standard reflections
3958 measured reflections	every 120 min
2017 independent reflections	intensity decay: 1.0%
1780 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
$R[F^2 > 2\sigma(F^2)]$ = 0.055	$w = 1/[\sigma^2(F_o^2) + (0.1016P)^2 + 1.9543P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.156	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.04	$\Delta\rho_{max}$ = 0.61 e Å^{-3}
2017 reflections	$\Delta\rho_{min}$ = −0.57 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: 0.0026 (3)
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}
C4	0.8787 (2)	0.4639 (4)	0.21953 (18)	0.0379 (7)
C5	0.7991 (2)	0.4206 (4)	0.27435 (17)	0.0363 (7)
C8	0.7702 (2)	0.2521 (4)	0.38080 (17)	0.0376 (7)
C9	0.8146 (2)	0.1638 (4)	0.43387 (17)	0.0411 (7)
C10	0.7655 (3)	0.0913 (4)	0.48874 (18)	0.0455 (8)
H10	0.7961	0.0307	0.5233	0.055*
C11	0.6702 (3)	0.1103 (4)	0.49132 (17)	0.0441 (8)
C12	0.6239 (2)	0.1961 (5)	0.4390 (2)	0.0473 (8)
H12	0.5592	0.2064	0.4410	0.057*
C13	0.6742 (2)	0.2669 (4)	0.38368 (18)	0.0444 (8)
H13	0.6431	0.3245	0.3483	0.053*
N7	0.8264 (2)	0.3205 (4)	0.32653 (15)	0.0432 (7)
H7N	0.8846	0.2955	0.3267	0.052*
O6	0.72271 (18)	0.4723 (3)	0.26554 (15)	0.0549 (7)
Cl1	0.85165 (7)	0.63777 (11)	0.17582 (6)	0.0615 (4)
Cl2	0.88387 (7)	0.31299 (12)	0.15463 (5)	0.0555 (3)
Cl3	0.98924 (6)	0.48027 (15)	0.26205 (5)	0.0634 (4)
Cl14	0.93496 (6)	0.13974 (14)	0.43103 (6)	0.0628 (4)
Cl15	0.60865 (8)	0.02480 (14)	0.56195 (5)	0.0654 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C4	0.0413 (16)	0.0405 (17)	0.0317 (15)	0.0016 (13)	0.0019 (12)	0.0003 (14)
C5	0.0360 (15)	0.0407 (15)	0.0323 (15)	−0.0028 (13)	0.0027 (12)	−0.0064 (13)
C8	0.0427 (16)	0.0431 (16)	0.0269 (14)	−0.0022 (13)	0.0021 (12)	−0.0029 (13)
C9	0.0388 (16)	0.0503 (18)	0.0343 (16)	−0.0006 (14)	−0.0053 (12)	−0.0033 (14)
C10	0.056 (2)	0.0504 (18)	0.0299 (16)	−0.0004 (16)	−0.0030 (14)	0.0018 (14)
C11	0.0546 (19)	0.0504 (18)	0.0273 (15)	−0.0053 (15)	0.0073 (13)	−0.0009 (14)
C12	0.0396 (16)	0.055 (2)	0.047 (2)	−0.0009 (15)	0.0063 (14)	0.0037 (17)
C13	0.0449 (16)	0.0541 (19)	0.0341 (15)	0.0038 (15)	0.0027 (13)	0.0058 (15)
N7	0.0348 (13)	0.0548 (17)	0.0400 (15)	0.0028 (12)	0.0055 (11)	0.0101 (13)

O6	0.0440 (14)	0.0669 (17)	0.0539 (16)	0.0076 (12)	0.0069 (11)	0.0172 (13)
Cl1	0.0623 (6)	0.0503 (6)	0.0718 (7)	0.0066 (4)	0.0147 (5)	0.0210 (5)
Cl2	0.0710 (6)	0.0582 (6)	0.0373 (5)	0.0051 (4)	0.0075 (4)	−0.0117 (4)
Cl3	0.0425 (5)	0.0980 (8)	0.0499 (6)	−0.0173 (5)	−0.0058 (4)	0.0079 (5)
Cl14	0.0406 (5)	0.0869 (8)	0.0610 (6)	0.0034 (4)	−0.0072 (4)	0.0154 (5)
Cl15	0.0772 (7)	0.0718 (7)	0.0473 (6)	−0.0046 (5)	0.0198 (4)	0.0156 (5)

Geometric parameters (Å, °)

C4—C5	1.564 (4)	C9—Cl14	1.735 (3)
C4—Cl1	1.744 (3)	C10—C11	1.374 (5)
C4—Cl2	1.764 (3)	C10—H10	0.9300
C4—Cl3	1.769 (3)	C11—C12	1.379 (5)
C5—O6	1.190 (4)	C11—Cl15	1.730 (3)
C5—N7	1.347 (4)	C12—C13	1.385 (5)
C8—C13	1.381 (5)	C12—H12	0.9300
C8—C9	1.389 (5)	C13—H13	0.9300
C8—N7	1.408 (4)	N7—H7N	0.8600
C9—C10	1.377 (5)		
C5—C4—Cl1	109.8 (2)	C11—C10—C9	118.5 (3)
C5—C4—Cl2	106.7 (2)	C11—C10—H10	120.7
Cl1—C4—Cl2	109.54 (18)	C9—C10—H10	120.7
C5—C4—Cl3	112.8 (2)	C10—C11—C12	121.1 (3)
Cl1—C4—Cl3	109.38 (18)	C10—C11—Cl15	118.7 (3)
Cl2—C4—Cl3	108.55 (18)	C12—C11—Cl15	120.2 (3)
O6—C5—N7	127.1 (3)	C11—C12—C13	119.7 (3)
O6—C5—C4	119.5 (3)	C11—C12—H12	120.1
N7—C5—C4	113.4 (3)	C13—C12—H12	120.1
C13—C8—C9	118.6 (3)	C8—C13—C12	120.3 (3)
C13—C8—N7	123.8 (3)	C8—C13—H13	119.9
C9—C8—N7	117.6 (3)	C12—C13—H13	119.9
C10—C9—C8	121.8 (3)	C5—N7—C8	127.1 (3)
C10—C9—Cl14	118.3 (3)	C5—N7—H7N	116.4
C8—C9—Cl14	119.9 (3)	C8—N7—H7N	116.4
Cl1—C4—C5—O6	23.0 (4)	C9—C10—C11—C12	2.0 (6)
Cl2—C4—C5—O6	−95.6 (3)	C9—C10—C11—Cl15	−177.8 (3)
Cl3—C4—C5—O6	145.3 (3)	C10—C11—C12—C13	−1.2 (6)
Cl1—C4—C5—N7	−159.3 (2)	Cl15—C11—C12—C13	178.6 (3)
Cl2—C4—C5—N7	82.1 (3)	C9—C8—C13—C12	0.7 (5)
Cl3—C4—C5—N7	−37.0 (3)	N7—C8—C13—C12	−179.9 (3)
C13—C8—C9—C10	0.0 (5)	C11—C12—C13—C8	−0.2 (6)
N7—C8—C9—C10	−179.4 (3)	O6—C5—N7—C8	1.7 (6)
C13—C8—C9—Cl14	178.8 (3)	C4—C5—N7—C8	−175.7 (3)
N7—C8—C9—Cl14	−0.6 (4)	C13—C8—N7—C5	7.0 (5)
C8—C9—C10—C11	−1.4 (5)	C9—C8—N7—C5	−173.7 (3)
Cl14—C9—C10—C11	179.8 (3)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N7—H7N···Cl3	0.86	2.49	2.953 (3)	115
N7—H7N···Cl14	0.86	2.44	2.916 (3)	115

Fig. 1

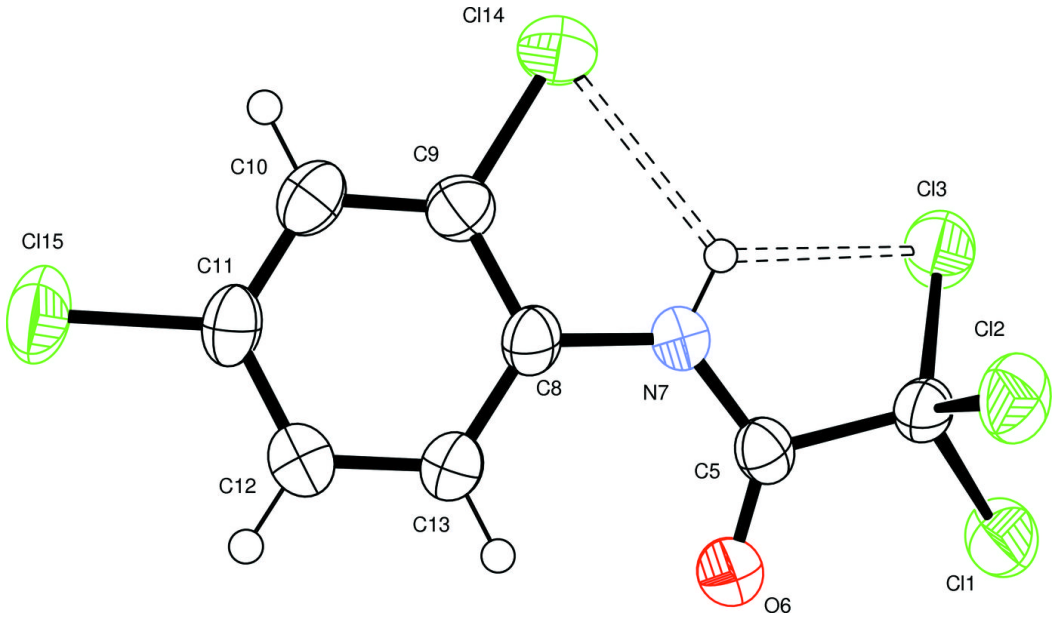

Fig. 2