Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,2,2-Trichloro-*N*-(3-nitrophenyl)-acetamide

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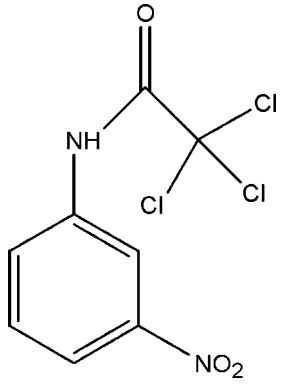
Received 27 July 2012; accepted 24 August 2012

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.049; wR factor = 0.144; data-to-parameter ratio = 17.5.

In the title compound, $C_8H_5Cl_3N_2O_3$, the dihedral angle between the nitrophenyl ring and the acetamide group is 5.47 (6)°. In the crystal, N—H···O and C—H···O hydrogen bonds link the molecules into chains running parallel to the b axis.

Related literature

For background to acetamides, see: Khan *et al.* (2010); Tahir & Shad (2011). For a related structure, see: Rosli *et al.* (2007). For hydrogen-bond motifs, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_8H_5Cl_3N_2O_3$ M_r = 283.49
Orthorhombic, *Pbca*
a = 11.5164 (8) Å
b = 10.1427 (5) Å
c = 19.9054 (11) Å
V = 2325.1 (2) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.78 mm^{-1}
T = 296 K
0.20 × 0.18 × 0.18 mm

Data collection

Bruker SMART APEX CCD detector diffractometer
Absorption correction: multi-scan (*SAINT-Plus*; Bruker, 1998)
T_{min} = 0.860, T_{max} = 0.872
8739 measured reflections
2532 independent reflections
1713 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.144
S = 1.07
2532 reflections
145 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 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
C4—H4···O1^{i}	0.93	2.59	3.345 (4)	138
N2—H2N···O1^{i}	0.86	2.15	2.990 (3)	164

Symmetry code: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, z$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1998) (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *CAMERON* (Watkin *et al.*, 1993); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

ANP is thankful to Manipal Institute of Technology, Manipal University and Mohamed Ziaulla, Bangalore University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2576).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker. (1998). *SMART* and *SAINT-Plus*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Khan, F. N., Roopan, S. M., Malathi, N., Hathwar, V. R. & Akkurt, M. (2010). *Acta Cryst.* E**66**, o2043–o2044.
Rosli, M. M., Karthikeyan, M. S., Fun, H.-K., Razak, I. A. & Patil, P. S. (2007). *Acta Cryst.* E**63**, o67–o68.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Tahir, M. N. & Shad, H. A. (2011). *Acta Cryst.* E**67**, o443.
Watkin, D. J., Prout, C. K. & Pearce, L. J. (1993). *CAMERON*. Chemical Crystallography Laboratory, University of Oxford, England.

supporting information

Acta Cryst. (2012). E**68**, o2832 [https://doi.org/10.1107/S1600536812036732]

2,2,2-Trichloro-*N*-(3-nitrophenyl)acetamide

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S1. Comment

In view of earlier studies and interest owing to the biological activity of acetamide (Khan *et al.*, 2010; Tahir & Shad, 2011), we report herein the crystal structure of the title compound. In the title compound, the nitro phenyl ring makes a dihedral angle of 5.47 (6)° with acetamide group. Bond lengths and angles are within the normal ranges and are comparable with a related structure (Rosli *et al.*, 2007). In the crystal, an S(6) ring motif (Bernstein *et al.*, 1995) is formed *via* intramolecular C8—H8A···O1 hydrogen bond (Table 1). The C—H···O and C—H···N hydrogen bonding intractions (Table 1 and Figure 2) result in bifurcated bonds and link the molecules into chains along the *b*-axis.

S2. Experimental

3-Nitroaniline (1.0 g, 0.0072 mmol) was dissolved in 2*M* hydrochloric acid (5.0 ml), and added a little crushed ice. A solution of hydrated sodium acetate (5.0 g) in 25 ml of water was introduced, followed by trichloroacetic anhydride (4.5 g, 0.01457 mol). The mixture was shaken in the cold until the smell of trichloroacetic anhydride disappeared. The title compound was collected by filtration and recrystallized from an aqueous ethanol (75%) solution; yield: 1.52 g (75%), m.p. 376.15–378.15 K.

S3. Refinement

The H atoms were placed at calculated positions in the riding model approximation with N—H = 0.86 and C—H = 0.93 Å, and U_{iso}(H) = 1.2U_{eq}(N/C).

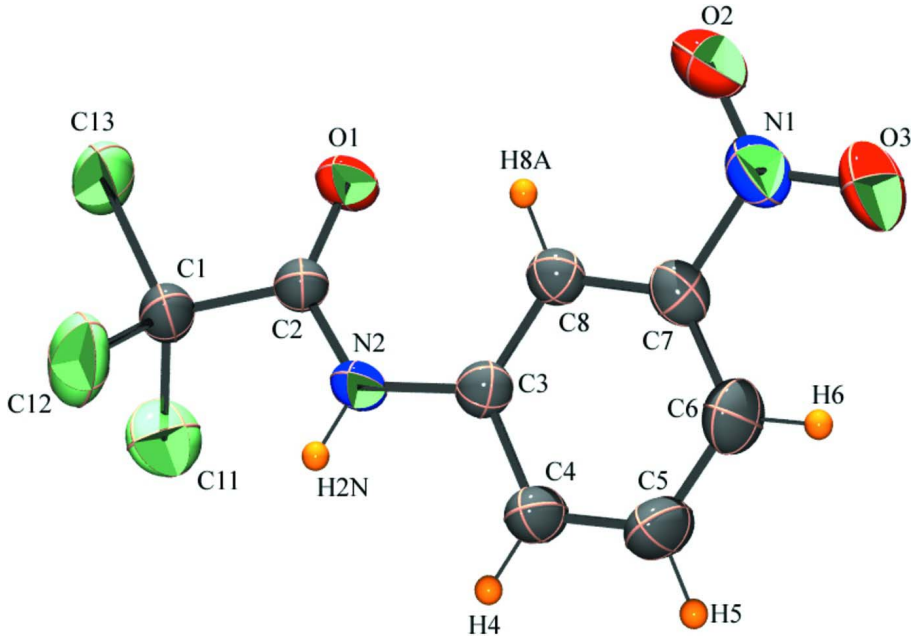

Figure 1

ORTEP (Farrugia, 1997) view of the title compound, showing 50% probability ellipsoids and the atom numbering scheme.

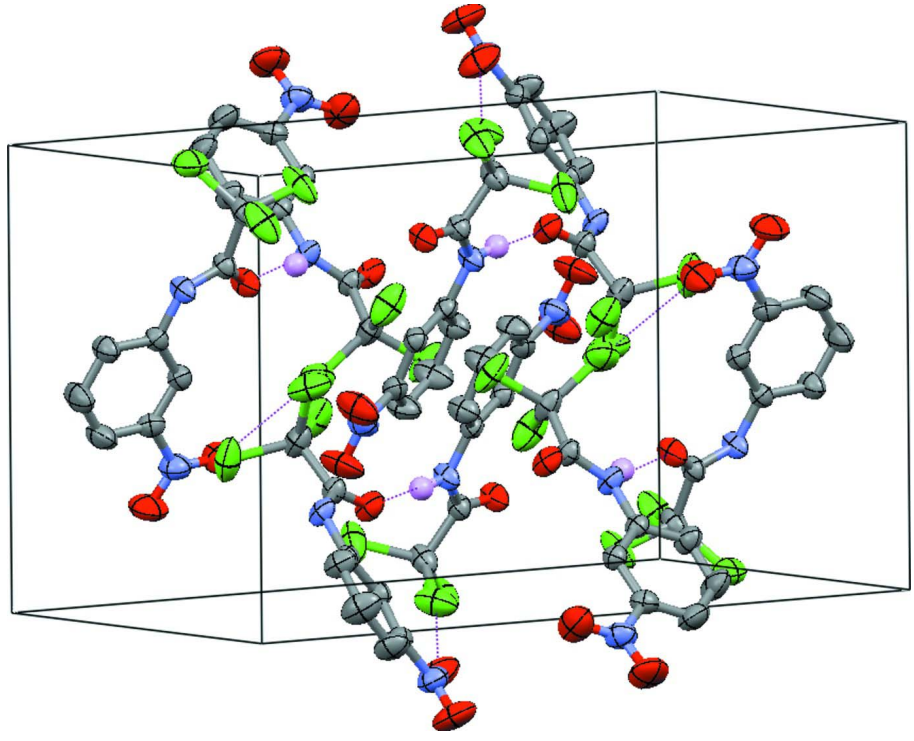

Figure 2

A unit cell packing of the title compound showing intermolecular interactions with dotted lines. H-atoms not involved in hydrogen bonding have been excluded.

2,2,2-Trichloro-*N*-(3-nitrophenyl)acetamide

Crystal data

$C_8H_5Cl_3N_2O_3$
$M_r = 283.49$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 11.5164$ (8) Å
$b = 10.1427$ (5) Å
$c = 19.9054$ (11) Å
$V = 2325.1$ (2) Å^3
$Z = 8$
$F(000) = 1136$
$D_x = 1.620$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2532 reflections
$\theta = 2.1$–$27.0°$
$\mu = 0.78$ mm^{-1}
$T = 296$ K
Block, yellow
0.20 × 0.18 × 0.18 mm

Data collection

Bruker SMART APEX CCD detector diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SAINT-Plus*; Bruker, 1998)
$T_{min} = 0.860$, $T_{max} = 0.872$
8739 measured reflections
2532 independent reflections
1713 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 27.0°$, $\theta_{min} = 2.1°$
$h = -5 \rightarrow 14$
$k = -10 \rightarrow 12$
$l = -14 \rightarrow 25$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.144$
$S = 1.07$
2532 reflections
145 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0695P)^2 + 0.9222P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.46$ e Å^{-3}
$\Delta\rho_{min} = -0.37$ e Å^{-3}

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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	0.8599 (3)	0.8973 (3)	0.29007 (15)	0.0522 (8)
C2	0.7513 (2)	0.9294 (2)	0.33324 (13)	0.0390 (6)
C3	0.5939 (2)	0.8197 (2)	0.39617 (13)	0.0367 (6)
C4	0.5685 (3)	0.7018 (3)	0.42914 (16)	0.0520 (7)
H4	0.6196	0.6310	0.4260	0.062*

C5	0.4687 (3)	0.6897 (3)	0.4662 (2)	0.0705 (10)
H5	0.4522	0.6103	0.4876	0.085*
C6	0.3927 (3)	0.7935 (3)	0.47228 (18)	0.0621 (9)
H6	0.3243	0.7852	0.4968	0.074*
C7	0.4209 (2)	0.9099 (3)	0.44096 (14)	0.0429 (6)
C8	0.5197 (2)	0.9263 (2)	0.40254 (13)	0.0387 (6)
H8A	0.5358	1.0063	0.3817	0.046*
Cl1	0.82157 (11)	0.79451 (9)	0.22201 (5)	0.0843 (4)
Cl3	0.92258 (9)	1.04369 (8)	0.25994 (5)	0.0801 (4)
Cl2	0.96357 (8)	0.81360 (10)	0.34058 (6)	0.0828 (3)
N1	0.3404 (2)	1.0223 (3)	0.44728 (15)	0.0580 (7)
N2	0.6956 (2)	0.82155 (19)	0.35614 (12)	0.0433 (6)
H2N	0.7247	0.7464	0.3454	0.052*
O1	0.72365 (18)	1.04229 (16)	0.34446 (11)	0.0524 (5)
O2	0.3623 (2)	1.1225 (2)	0.41711 (16)	0.0858 (8)
O3	0.2570 (2)	1.0101 (3)	0.48485 (16)	0.0900 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0626 (19)	0.0350 (14)	0.0589 (18)	−0.0020 (13)	0.0223 (15)	0.0010 (12)
C2	0.0444 (14)	0.0307 (12)	0.0418 (14)	−0.0010 (11)	0.0055 (13)	0.0007 (10)
C3	0.0383 (14)	0.0318 (12)	0.0400 (13)	−0.0053 (10)	−0.0002 (12)	−0.0019 (10)
C4	0.0499 (17)	0.0353 (14)	0.071 (2)	0.0007 (12)	0.0134 (16)	0.0083 (13)
C5	0.064 (2)	0.0461 (18)	0.101 (3)	−0.0008 (15)	0.027 (2)	0.0226 (17)
C6	0.0453 (17)	0.0588 (19)	0.082 (2)	−0.0057 (14)	0.0193 (17)	0.0078 (16)
C7	0.0368 (14)	0.0433 (14)	0.0485 (15)	0.0011 (11)	−0.0027 (13)	−0.0063 (12)
C8	0.0412 (14)	0.0332 (13)	0.0416 (14)	−0.0015 (10)	−0.0005 (12)	−0.0017 (10)
Cl1	0.1194 (9)	0.0689 (6)	0.0645 (6)	−0.0233 (5)	0.0383 (6)	−0.0211 (4)
Cl3	0.0991 (8)	0.0439 (4)	0.0973 (7)	−0.0162 (4)	0.0522 (6)	0.0022 (4)
Cl2	0.0527 (5)	0.0816 (6)	0.1141 (8)	0.0127 (4)	0.0146 (5)	0.0168 (6)
N1	0.0462 (15)	0.0550 (16)	0.0728 (18)	0.0068 (12)	0.0062 (14)	−0.0033 (13)
N2	0.0482 (13)	0.0234 (10)	0.0584 (14)	0.0020 (9)	0.0148 (11)	0.0000 (9)
O1	0.0560 (12)	0.0243 (9)	0.0767 (14)	−0.0014 (8)	0.0169 (11)	−0.0020 (8)
O2	0.0786 (18)	0.0568 (15)	0.122 (2)	0.0245 (13)	0.0300 (16)	0.0152 (15)
O3	0.0544 (15)	0.0901 (18)	0.125 (2)	0.0202 (13)	0.0345 (16)	0.0105 (17)

Geometric parameters (Å, º)

C1—C2	1.552 (4)	C5—C6	1.374 (4)
C1—Cl3	1.756 (3)	C5—H5	0.9300
C1—Cl1	1.766 (3)	C6—C7	1.374 (4)
C1—Cl2	1.777 (3)	C6—H6	0.9300
C2—O1	1.209 (3)	C7—C8	1.380 (4)
C2—N2	1.348 (3)	C7—N1	1.476 (4)
C3—C8	1.385 (3)	C8—H8A	0.9300
C3—C4	1.395 (3)	N1—O2	1.207 (3)
C3—N2	1.417 (3)	N1—O3	1.223 (4)

C4—C5	1.372 (4)	N2—H2N	0.8600
C4—H4	0.9300		
C2—C1—Cl3	110.05 (18)	C6—C5—H5	119.6
C2—C1—Cl1	110.3 (2)	C7—C6—C5	117.9 (3)
Cl3—C1—Cl1	109.88 (16)	C7—C6—H6	121.1
C2—C1—Cl2	109.17 (19)	C5—C6—H6	121.1
Cl3—C1—Cl2	108.75 (18)	C6—C7—C8	123.4 (3)
Cl1—C1—Cl2	108.65 (15)	C6—C7—N1	118.5 (3)
O1—C2—N2	125.5 (2)	C8—C7—N1	118.1 (2)
O1—C2—C1	120.9 (2)	C7—C8—C3	117.7 (2)
N2—C2—C1	113.6 (2)	C7—C8—H8A	121.1
C8—C3—C4	119.8 (2)	C3—C8—H8A	121.1
C8—C3—N2	123.5 (2)	O2—N1—O3	123.6 (3)
C4—C3—N2	116.7 (2)	O2—N1—C7	118.5 (3)
C5—C4—C3	120.4 (3)	O3—N1—C7	117.8 (3)
C5—C4—H4	119.8	C2—N2—C3	126.5 (2)
C3—C4—H4	119.8	C2—N2—H2N	116.8
C4—C5—C6	120.8 (3)	C3—N2—H2N	116.8
C4—C5—H5	119.6		
Cl3—C1—C2—O1	−3.2 (4)	C6—C7—C8—C3	−0.6 (4)
Cl1—C1—C2—O1	−124.6 (3)	N1—C7—C8—C3	−179.1 (2)
Cl2—C1—C2—O1	116.1 (3)	C4—C3—C8—C7	−1.2 (4)
Cl3—C1—C2—N2	177.6 (2)	N2—C3—C8—C7	177.4 (2)
Cl1—C1—C2—N2	56.2 (3)	C6—C7—N1—O2	−176.5 (3)
Cl2—C1—C2—N2	−63.1 (3)	C8—C7—N1—O2	2.1 (4)
C8—C3—C4—C5	1.9 (5)	C6—C7—N1—O3	6.1 (4)
N2—C3—C4—C5	−176.8 (3)	C8—C7—N1—O3	−175.3 (3)
C3—C4—C5—C6	−0.8 (6)	O1—C2—N2—C3	1.3 (5)
C4—C5—C6—C7	−1.0 (6)	C1—C2—N2—C3	−179.5 (3)
C5—C6—C7—C8	1.8 (5)	C8—C3—N2—C2	18.2 (4)
C5—C6—C7—N1	−179.7 (3)	C4—C3—N2—C2	−163.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8*A*···O1	0.93	2.32	2.870 (3)	117
C4—H4···O1^{i}	0.93	2.59	3.345 (4)	138
N2—H2*N*···O1^{i}	0.86	2.15	2.990 (3)	164

Symmetry code: (i) −*x*+3/2, *y*−1/2, *z*.