

2,2,2-Tribromo-N-(3-chlorophenyl)-acetamide

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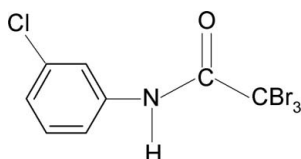
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Key indicators: single-crystal X-ray study; $T = 299$ K; mean $\sigma(\text{C}-\text{C}) = 0.018$ Å; R factor = 0.086; wR factor = 0.387; data-to-parameter ratio = 16.6.

In the title compound, $\text{C}_8\text{H}_5\text{Br}_3\text{ClNO}$, the conformation of the N—H bond is *anti* to the 3-chloro substituent in the benzene ring. An intramolecular N—H \cdots Br hydrogen bond occurs. In the crystal, molecules are packed into infinite chains in the *a*-axis direction by N—H \cdots O hydrogen bonds.

Related literature

For the preparation of the title compound, see: Gowda *et al.* (2003). For background and related structures, see: Brown (1966); Gowda *et al.* (2008, 2009, 2010).



Experimental

Crystal data

$\text{C}_8\text{H}_5\text{Br}_3\text{ClNO}$
 $M_r = 406.31$
Orthorhombic, *Pbca*
 $a = 12.803$ (1) Å
 $b = 9.146$ (1) Å
 $c = 20.221$ (3) Å

$V = 2367.8$ (5) Å³
 $Z = 8$
Cu $K\alpha$ radiation
 $\mu = 14.47$ mm⁻¹
 $T = 299$ K
0.53 × 0.33 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.049$, $T_{\max} = 0.123$
3870 measured reflections

2114 independent reflections
1646 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.110$
3 standard reflections every 120 min
intensity decay: 1.5%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.086$
 $wR(F^2) = 0.387$
 $S = 1.59$
2114 reflections

127 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 2.07$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.56$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1N \cdots O1 ¹	0.86	2.20	3.032 (13)	162
N1—H1N \cdots Br3	0.86	2.84	3.177 (9)	105

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

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, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); 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: FL2301).

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supplementary materials

Acta Cryst. (2010). E66, o1140 [doi:10.1107/S160053681001411X]

2,2,2-Tribromo-*N*-(3-chlorophenyl)acetamide

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Comment

The structure of (I), was determined as a part of our ongoing study of the effect of ring and side chain substituents on the crystal structures of *N*-aromatic amides (Gowda *et al.*, 2008, 2009, 2010). In (I) the conformation of the N—H bond is *anti* to the 3-chloro substituent in the benzene ring (Fig. 1), similar to that observed in *N*-(3-chlorophenyl)acetamide (II)(Gowda *et al.*, 2008), and that between the N—H bond and the 3-methyl group in *N*-(3-methylphenyl)2,2,2-tribromoacetamide (III)(Gowda *et al.*, 2009), but contrary to the *syn* conformation observed between the N—H bond and the 2-Chloro group in *N*-(2-chlorophenyl)2,2,2-tribromoacetamide (IV) (Gowda *et al.*, 2010).

Further, the conformation of the N—H bond in (I) is *anti* to the C=O bond in the side chain, similar to that observed in *N*-(phenyl)2,2,2-tribromoacetamide, (II), (III) and (IV) (Gowda *et al.*, 2008, 2009, 2010) and other amides (Brown, 1966).

The structure of (I) shows both intramolecular N—H \cdots Br and intermolecular N—H \cdots O H-bonding. A packing diagram (Fig. 2) illustrates the N1—H1N \cdots O1 hydrogen bonds (Table 1) involved in the formation of molecular chains along the *a*-axis of the unit cell.

Experimental

The title compound was prepared from 3-chloroaniline, tribromoacetic acid and phosphorylchloride according to the literature method (Gowda *et al.*, 2003). The purity of the compound was checked by determining its melting point. It was further characterized by recording its infrared spectra. Rod like colourless single crystals of the title compound used for X-ray diffraction studies were obtained by a slow evaporation of its ethanolic solution at room temperature.

Refinement

The H atoms were positioned with idealized geometry using a riding model [N—H = 0.86 Å, C—H = 0.93 Å] and were refined with a riding model conith isotropic displacement parameters (set to 1.2 times of the U_{eq} of the parent atom).

The residual electron-density features are located in the region of Br1 and Br2. The highest peak is 0.98 Å from Br1 and the deepest hole is 1.39 Å from Br2.

Figures

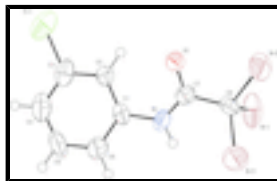


Fig. 1. Molecular structure of (I), showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented as small spheres of arbitrary radii.

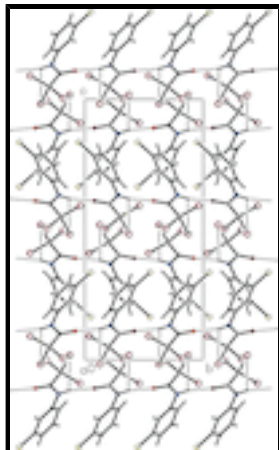


Fig. 2. Molecular packing of (I) with hydrogen bonds shown as dashed lines.

2,2,2-Tribromo-N-(3-chlorophenyl)acetamide

Crystal data

$C_8H_5Br_3ClNO$

$M_r = 406.31$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 12.803 (1) \text{ \AA}$

$b = 9.146 (1) \text{ \AA}$

$c = 20.221 (3) \text{ \AA}$

$V = 2367.8 (5) \text{ \AA}^3$

$Z = 8$

$F(000) = 1520$

$D_x = 2.280 \text{ Mg m}^{-3}$

Cu $K\alpha$ radiation, $\lambda = 1.54180 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 4.4\text{--}20.5^\circ$

$\mu = 14.47 \text{ mm}^{-1}$

$T = 299 \text{ K}$

Rod, colourless

$0.53 \times 0.33 \times 0.25 \text{ mm}$

Data collection

Enraf-Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube
graphite

$\omega/2\theta$ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.049$, $T_{\max} = 0.123$

3870 measured reflections

2114 independent reflections

1646 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.110$

$\theta_{\max} = 67.0^\circ$, $\theta_{\min} = 4.4^\circ$

$h = -15 \rightarrow 11$

$k = -10 \rightarrow 0$

$l = -24 \rightarrow 0$

3 standard reflections every 120 min

intensity decay: 1.5%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.086$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.387$	H-atom parameters constrained
$S = 1.59$	$w = 1/[\sigma^2(F_o^2) + (0.2P)^2]$
2114 reflections	where $P = (F_o^2 + 2F_c^2)/3$
127 parameters	$(\Delta/\sigma)_{\max} = 0.006$
0 restraints	$\Delta\rho_{\max} = 2.07 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\min} = -1.56 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.8142 (8)	0.2739 (12)	0.3141 (5)	0.042 (2)
C2	0.7763 (8)	0.3768 (12)	0.2709 (6)	0.043 (2)
H2	0.7101	0.4167	0.2769	0.051*
C3	0.8383 (13)	0.4204 (15)	0.2184 (6)	0.057 (3)
C4	0.9361 (13)	0.3617 (16)	0.2099 (8)	0.071 (4)
H4	0.9773	0.3902	0.1743	0.085*
C5	0.9715 (11)	0.2635 (18)	0.2533 (9)	0.077 (5)
H5	1.0374	0.2231	0.2467	0.092*
C6	0.9141 (11)	0.2199 (13)	0.3074 (8)	0.059 (3)
H6	0.9419	0.1559	0.3385	0.071*
C7	0.6833 (9)	0.2898 (11)	0.4008 (5)	0.042 (2)
C8	0.6221 (8)	0.2020 (11)	0.4511 (6)	0.043 (2)
Br1	0.55384 (15)	0.04007 (19)	0.40907 (9)	0.0787 (8)
Br2	0.52069 (17)	0.31822 (18)	0.49631 (11)	0.0893 (9)
Br3	0.71709 (15)	0.1263 (3)	0.51772 (8)	0.0856 (8)
Cl1	0.7921 (4)	0.5505 (5)	0.16439 (19)	0.0817 (13)
N1	0.7560 (8)	0.2201 (11)	0.3684 (5)	0.050 (2)
H1N	0.7701	0.1327	0.3814	0.060*
O1	0.6555 (7)	0.4164 (8)	0.3902 (4)	0.0494 (19)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.032 (5)	0.046 (5)	0.047 (6)	-0.008 (4)	0.008 (4)	-0.005 (5)
C2	0.036 (5)	0.045 (5)	0.048 (6)	-0.006 (4)	0.000 (4)	0.005 (4)

supplementary materials

C3	0.069 (8)	0.065 (7)	0.039 (5)	-0.018 (6)	0.006 (5)	0.001 (5)
C4	0.082 (10)	0.051 (7)	0.079 (10)	-0.009 (7)	0.041 (8)	-0.003 (7)
C5	0.045 (8)	0.070 (9)	0.116 (12)	-0.003 (7)	0.040 (8)	0.004 (10)
C6	0.060 (7)	0.040 (5)	0.076 (8)	0.003 (5)	0.017 (6)	0.004 (6)
C7	0.044 (6)	0.037 (5)	0.044 (5)	-0.002 (4)	-0.001 (4)	0.000 (4)
C8	0.032 (5)	0.037 (5)	0.061 (6)	0.006 (4)	0.004 (5)	-0.005 (4)
Br1	0.0826 (13)	0.0758 (12)	0.0776 (12)	-0.0431 (9)	0.0248 (8)	-0.0149 (8)
Br2	0.0953 (15)	0.0610 (12)	0.1114 (16)	0.0226 (9)	0.0628 (12)	0.0087 (9)
Br3	0.0700 (12)	0.1248 (18)	0.0619 (12)	0.0134 (10)	-0.0027 (7)	0.0327 (10)
Cl1	0.117 (3)	0.079 (2)	0.0498 (19)	-0.006 (2)	0.0019 (18)	0.0161 (16)
N1	0.056 (6)	0.041 (4)	0.052 (5)	0.000 (4)	0.016 (5)	0.015 (4)
O1	0.042 (4)	0.037 (3)	0.069 (5)	0.000 (3)	0.008 (4)	0.009 (3)

Geometric parameters (Å, °)

C1—C2	1.372 (16)	C5—H5	0.9300
C1—C6	1.378 (17)	C6—H6	0.9300
C1—N1	1.416 (13)	C7—O1	1.229 (14)
C2—C3	1.385 (16)	C7—N1	1.305 (16)
C2—H2	0.9300	C7—C8	1.515 (15)
C3—C4	1.37 (2)	C8—Br2	1.911 (10)
C3—Cl1	1.720 (15)	C8—Br1	1.918 (11)
C4—C5	1.33 (2)	C8—Br3	1.943 (11)
C4—H4	0.9300	N1—H1N	0.8600
C5—C6	1.377 (18)		
C2—C1—C6	120.8 (10)	C5—C6—C1	118.0 (14)
C2—C1—N1	123.1 (10)	C5—C6—H6	121.0
C6—C1—N1	116.1 (11)	C1—C6—H6	121.0
C1—C2—C3	118.8 (11)	O1—C7—N1	125.4 (10)
C1—C2—H2	120.6	O1—C7—C8	117.8 (10)
C3—C2—H2	120.6	N1—C7—C8	116.5 (9)
C4—C3—C2	120.4 (13)	C7—C8—Br2	112.2 (7)
C4—C3—Cl1	120.3 (10)	C7—C8—Br1	110.4 (8)
C2—C3—Cl1	119.3 (12)	Br2—C8—Br1	109.4 (5)
C5—C4—C3	119.4 (12)	C7—C8—Br3	109.3 (7)
C5—C4—H4	120.3	Br2—C8—Br3	107.0 (6)
C3—C4—H4	120.3	Br1—C8—Br3	108.5 (5)
C4—C5—C6	122.4 (14)	C7—N1—C1	126.5 (10)
C4—C5—H5	118.8	C7—N1—H1N	116.8
C6—C5—H5	118.8	C1—N1—H1N	116.8
C6—C1—C2—C3	3.2 (17)	O1—C7—C8—Br2	-6.9 (13)
N1—C1—C2—C3	-178.6 (11)	N1—C7—C8—Br2	178.6 (9)
C1—C2—C3—C4	-0.3 (19)	O1—C7—C8—Br1	115.4 (10)
C1—C2—C3—Cl1	-179.4 (9)	N1—C7—C8—Br1	-59.1 (12)
C2—C3—C4—C5	-1(2)	O1—C7—C8—Br3	-125.4 (9)
Cl1—C3—C4—C5	178.3 (13)	N1—C7—C8—Br3	60.1 (12)
C3—C4—C5—C6	-1(3)	O1—C7—N1—C1	-2(2)
C4—C5—C6—C1	4(2)	C8—C7—N1—C1	171.9 (11)
C2—C1—C6—C5	-5(2)	C2—C1—N1—C7	-28.2 (19)

N1—C1—C6—C5

176.7 (13)

C6—C1—N1—C7

150.0 (13)

Hydrogen-bond geometry (Å, °)

D—H···*A*

D—H

H···*A*

D···*A*

D—H···*A*

N1—H1N···O1ⁱ

0.86

2.20

3.032 (13)

162

N1—H1N···Br3

0.86

2.84

3.177 (9)

105

Symmetry codes: (i) $-x+3/2, y-1/2, z$.

Fig. 1

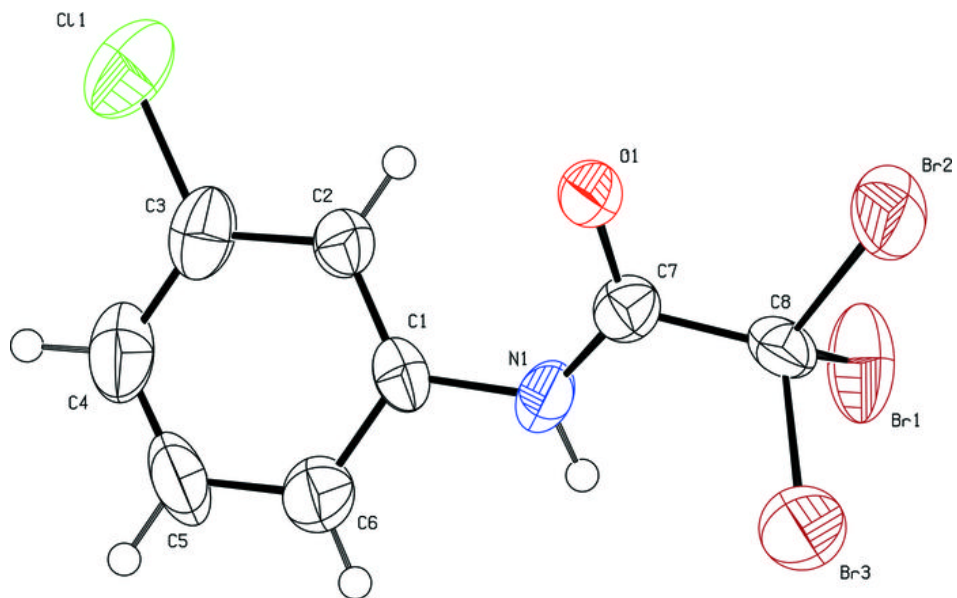


Fig. 2

