organic compounds

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

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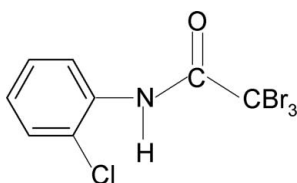Correspondence e-mail: gowdabt@yahoo.com

In the title compound, $C_8H_5Br_3ClNO$, the conformation of the N—H bond is *syn* to the 2-chloro substituent in the benzene ring. There are no classical intermolecular hydrogen bonds, but intramolecular N—H⋯Br and N—H⋯Cl contacts occur.

Related literature

For preparation of the title compound, see: Gowda *et al.* (2003). For background to our studies on the effect of the ring and the side-chain substituents on the crystal structures of *N*-aromatic amides, see: Gowda *et al.* (2007, 2009). For the conformations of other amides, see: Brown (1966).

Experimental

Crystal data

$C_8H_5Br_3ClNO$	V = 1134.98 (12) Å^3
M_r = 406.31	Z = 4
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation
a = 9.1947 (6) Å	μ = 10.86 mm^{-1}
b = 12.9645 (7) Å	T = 299 K
c = 9.5213 (6) Å	0.40 × 0.40 × 0.34 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	Diffraction, 2009)
	T_{min} = 0.098, T_{max} = 0.120
	4524 measured reflections
Absorption correction: multi-scan (*CrysAlis RED*; Oxford	1645 independent reflections
	1491 reflections with $I > 2\sigma(I)$
	R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.066	
S = 1.07	
1645 reflections	$\Delta\rho_{max}$ = 0.77 e Å^{-3}
131 parameters	$\Delta\rho_{min}$ = −0.56 e Å^{-3}
2 restraints	Absolute structure: Flack (1983), 416 Friedel pairs
	Flack parameter: 0.049 (18)

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1N⋯Br1	0.85 (3)	2.78 (8)	3.155 (6)	109 (6)
N1—H1N⋯Cl1	0.85 (3)	2.59 (7)	2.961 (5)	107 (5)

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2009); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2009); data reduction: *CrysAlis RED*; 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*.

References

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supporting information

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

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S1. Comment

As a part of studying the effect of the ring and the side chain substituents on the crystal structures of *N*-aromatic amides (Gowda *et al.*, 2007, 2009), in the present work, the structure of *N*-(2-chlorophenyl)2,2,2-tribromoacetamide (I) has been determined (Fig.1). The conformation of the N—H bond is *syn* to the 2-chloro substituent in the benzene ring, similar to that observed in *N*-(2-chlorophenyl)acetamide and *N*-(2-chlorophenyl)2,2,2-trichloroacetamide (Gowda *et al.*, 2007), but contrary to the *anti* conformation observed between the N—H bond and the 3-methyl group in *N*-(3-methylphenyl)2,2,2-tribromoacetamide (Gowda *et al.*, 2009). Further, the conformation of the N—H bond in the structure is *anti* to the C=O bond in the side chain, similar to that observed in *N*-(phenyl)2,2,2-tribromoacetamide (Gowda *et al.*, 2009) and other amides (Brown, 1966; Gowda *et al.*, 2007, 2009). The structure shows simultaneous N—H···Br and N—H···Cl intramolecular H-bonding. The packing diagram of the molecules is shown in Fig. 2.

S2. Experimental

The title compound was prepared from 2-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. 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.

S3. Refinement

The H atom of the NH group was located in a difference map and later restrained to the distance N—H = 0.86 (3) Å. The other H atoms were positioned with idealized geometry using a riding model [C—H = 0.93 Å]. All H atoms were refined with isotropic displacement parameters set to 1.2 times of the U_{eq} of the parent atom.
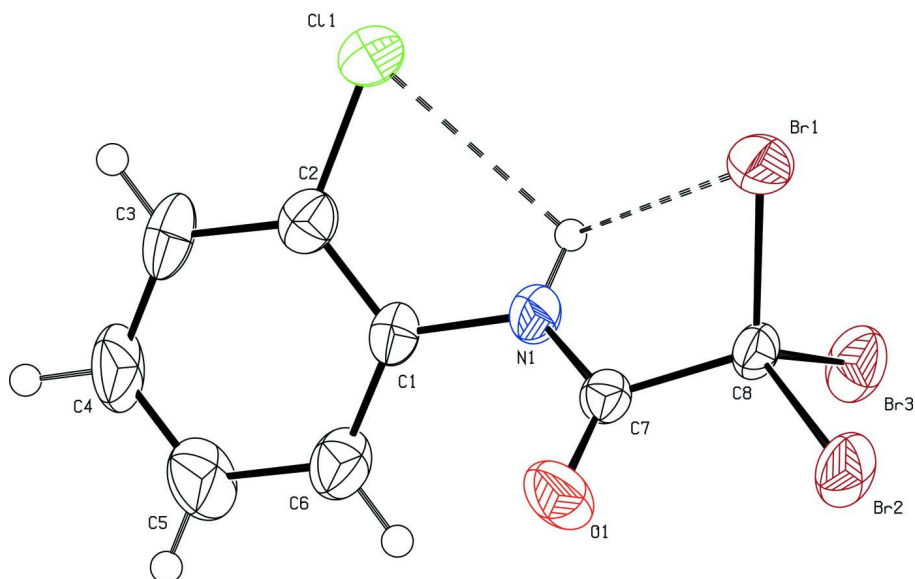
Figure 1

Molecular structure of the title compound, showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and the H atoms are represented as small spheres of arbitrary radii.
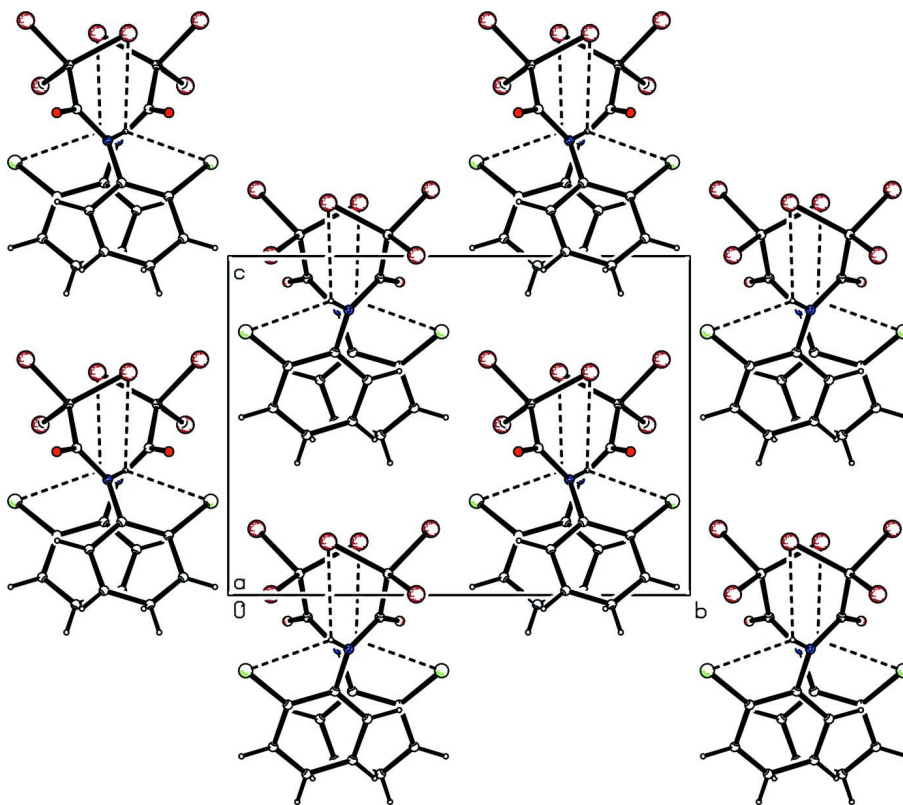
Figure 2

Molecular packing of the title compound with hydrogen bonds shown as dashed lines.

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

Crystal data

$C_8H_5Br_3ClNO$	$F(000) = 760$
$M_r = 406.31$	$D_x = 2.378$ Mg m^{-3}
Orthorhombic, *Pna*2_1	Mo *Kα* radiation, $\lambda = 0.71073$ Å
Hall symbol: P 2c -2n	Cell parameters from 3507 reflections
$a = 9.1947$ (6) Å	$\theta = 2.7–27.8°$
$b = 12.9645$ (7) Å	$\mu = 10.86$ mm^{-1}
$c = 9.5213$ (6) Å	$T = 299$ K
$V = 1134.98$ (12) Å^3	Rod, colourless
$Z = 4$	$0.40 \times 0.40 \times 0.34$ mm

Data collection

Oxford Diffraction Xcalibur diffractometer with a Sapphire CCD detector	4524 measured reflections
	1645 independent reflections
Radiation source: fine-focus sealed tube	1491 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.022$
Rotation method data acquisition using ω and φ scans.	$\theta_{max} = 26.4°$, $\theta_{min} = 2.7°$
	$h = -11 \rightarrow 11$
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009)	$k = -16 \rightarrow 15$
	$l = -11 \rightarrow 6$
$T_{min} = 0.098$, $T_{max} = 0.120$	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.027$	$w = 1/[\sigma^2(F_o^2) + (0.0357P)^2 + 1.2795P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.066$	
$S = 1.07$	$(\Delta/\sigma)_{max} = 0.004$
1645 reflections	$\Delta\rho_{max} = 0.77$ e Å^{-3}
131 parameters	$\Delta\rho_{min} = -0.56$ e Å^{-3}
2 restraints	Extinction correction: *SHELXL97* (Sheldrick, 2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Primary atom site location: structure-invariant direct methods	
	Extinction coefficient: 0.0072 (5)
Secondary atom site location: difference Fourier map	Absolute structure: Flack (1983), 416 Friedel pairs
Hydrogen site location: inferred from neighbouring sites	Absolute structure parameter: 0.049 (18)

Special details

Experimental. CrysAlis RED (Oxford Diffraction, 2009) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	$U_{iso}*/U_{eq}$
Br1	0.56049 (8)	0.28366 (5)	1.15778 (8)	0.0510 (2)
Br2	0.43394 (7)	0.06152 (5)	1.19542 (7)	0.0483 (2)
Br3	0.70743 (6)	0.09279 (5)	1.00260 (9)	0.0474 (2)
Cl1	0.5017 (2)	0.46215 (12)	0.7770 (2)	0.0536 (5)
O1	0.3140 (5)	0.1287 (4)	0.9233 (5)	0.0521 (13)
N1	0.4832 (5)	0.2390 (4)	0.8407 (6)	0.0366 (12)
H1N	0.554 (5)	0.277 (4)	0.866 (9)	0.044*
C1	0.4101 (6)	0.2689 (4)	0.7164 (7)	0.0311 (12)
C2	0.4114 (6)	0.3709 (4)	0.6763 (7)	0.0360 (14)
C3	0.3435 (8)	0.4026 (5)	0.5526 (7)	0.0475 (17)
H3	0.3476	0.4712	0.5244	0.057*
C4	0.2701 (8)	0.3305 (6)	0.4724 (8)	0.0573 (19)
H4	0.2235	0.3506	0.3901	0.069*
C5	0.2660 (9)	0.2308 (6)	0.5136 (9)	0.063 (2)
H5	0.2152	0.1829	0.4598	0.076*
C6	0.3362 (8)	0.1987 (5)	0.6347 (7)	0.0471 (17)
H6	0.3335	0.1296	0.6607	0.057*
C7	0.4282 (6)	0.1716 (4)	0.9346 (6)	0.0278 (12)
C8	0.5253 (6)	0.1542 (4)	1.0638 (7)	0.0288 (12)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0744 (5)	0.0373 (3)	0.0411 (4)	−0.0059 (3)	−0.0169 (4)	−0.0036 (3)
Br2	0.0476 (4)	0.0509 (4)	0.0464 (4)	−0.0058 (3)	0.0000 (3)	0.0248 (3)
Br3	0.0328 (3)	0.0471 (4)	0.0623 (5)	0.0097 (3)	0.0018 (3)	0.0139 (4)
Cl1	0.0620 (10)	0.0384 (8)	0.0604 (12)	−0.0142 (8)	−0.0155 (9)	0.0153 (8)
O1	0.047 (3)	0.067 (3)	0.043 (3)	−0.025 (2)	−0.009 (2)	0.011 (3)
N1	0.040 (3)	0.035 (3)	0.035 (3)	−0.007 (2)	−0.008 (2)	0.013 (3)
C1	0.035 (3)	0.032 (3)	0.026 (3)	0.008 (2)	0.000 (2)	0.004 (2)
C2	0.032 (3)	0.036 (3)	0.040 (4)	0.004 (2)	0.004 (3)	0.004 (3)
C3	0.057 (4)	0.052 (4)	0.033 (4)	0.018 (3)	0.004 (3)	0.018 (3)
C4	0.074 (5)	0.070 (5)	0.028 (4)	0.010 (4)	−0.017 (4)	0.012 (4)
C5	0.092 (5)	0.065 (5)	0.033 (4)	0.011 (4)	−0.020 (4)	−0.016 (4)
C6	0.068 (4)	0.039 (3)	0.034 (4)	0.011 (3)	−0.005 (4)	−0.002 (3)
C7	0.028 (3)	0.027 (3)	0.029 (3)	0.002 (2)	0.000 (2)	0.000 (2)
C8	0.032 (3)	0.027 (3)	0.027 (3)	0.001 (2)	−0.001 (2)	0.008 (2)

Geometric parameters (Å, º)

Br1—C8	1.929 (6)	C2—C3	1.395 (9)
Br2—C8	1.929 (6)	C3—C4	1.383 (10)
Br3—C8	1.944 (6)	C3—H3	0.9300
Cl1—C2	1.735 (7)	C4—C5	1.351 (11)
O1—C7	1.193 (6)	C4—H4	0.9300

N1—C7	1.349 (7)	C5—C6	1.385 (10)
N1—C1	1.415 (8)	C5—H5	0.9300
N1—H1N	0.85 (3)	C6—H6	0.9300
C1—C6	1.377 (9)	C7—C8	1.537 (8)
C1—C2	1.376 (8)		
C7—N1—C1	123.6 (5)	C4—C5—C6	121.1 (7)
C7—N1—H1N	118 (6)	C4—C5—H5	119.5
C1—N1—H1N	116 (5)	C6—C5—H5	119.5
C6—C1—C2	118.8 (6)	C1—C6—C5	120.1 (6)
C6—C1—N1	121.7 (5)	C1—C6—H6	119.9
C2—C1—N1	119.4 (6)	C5—C6—H6	119.9
C1—C2—C3	120.9 (6)	O1—C7—N1	124.9 (6)
C1—C2—Cl1	120.4 (5)	O1—C7—C8	121.0 (5)
C3—C2—Cl1	118.7 (5)	N1—C7—C8	114.1 (4)
C4—C3—C2	119.0 (6)	C7—C8—Br1	110.0 (4)
C4—C3—H3	120.5	C7—C8—Br2	111.0 (4)
C2—C3—H3	120.5	Br1—C8—Br2	108.3 (3)
C5—C4—C3	120.0 (6)	C7—C8—Br3	108.7 (4)
C5—C4—H4	120.0	Br1—C8—Br3	110.5 (3)
C3—C4—H4	120.0	Br2—C8—Br3	108.3 (3)
C7—N1—C1—C6	−41.6 (9)	N1—C1—C6—C5	−179.9 (6)
C7—N1—C1—C2	137.9 (6)	C4—C5—C6—C1	0.9 (12)
C6—C1—C2—C3	−2.1 (9)	C1—N1—C7—O1	1.1 (9)
N1—C1—C2—C3	178.4 (6)	C1—N1—C7—C8	−177.9 (5)
C6—C1—C2—Cl1	179.4 (5)	O1—C7—C8—Br1	−121.3 (5)
N1—C1—C2—Cl1	−0.1 (8)	N1—C7—C8—Br1	57.7 (6)
C1—C2—C3—C4	2.2 (10)	O1—C7—C8—Br2	−1.5 (7)
Cl1—C2—C3—C4	−179.4 (5)	N1—C7—C8—Br2	177.5 (4)
C2—C3—C4—C5	−0.7 (11)	O1—C7—C8—Br3	117.5 (5)
C3—C4—C5—C6	−0.9 (12)	N1—C7—C8—Br3	−63.4 (5)
C2—C1—C6—C5	0.6 (10)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···Br1	0.85 (3)	2.78 (8)	3.155 (6)	109 (6)
N1—H1*N*···Cl1	0.85 (3)	2.59 (7)	2.961 (5)	107 (5)