# organic compounds

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

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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).
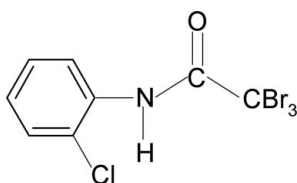


## Experimental

### 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 | $\mu = 10.86$ mm$^{-1}$ |
| $b = 12.9645$ (7) Å | $T = 299$ K |
| $c = 9.5213$ (6) Å | $0.40 \times 0.40 \times 0.34$ 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, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|---|---|---|---|
| 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

Brown, C. J. (1966). *Acta Cryst.* **21**, 442–445.
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Oxford Diffraction (2009). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Yarnton, England.
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**supplementary materials**

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

**B. T. Gowda, S. Foro, P. A. Suchetan** and **H. Fuess**

### 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.

### 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.

### 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.
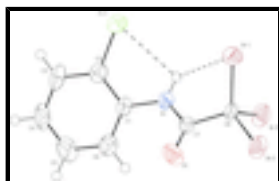
### Figures



Fig. 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.
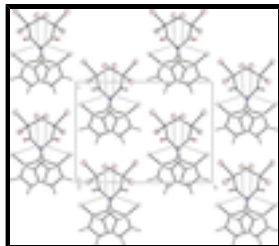
Fig. 2. Molecular packing of the title compound with hydrogen bonds shown as dashed lines.

**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, $Pna2_1$ | Mo $K\alpha$ 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 | 1645 independent reflections |
| Radiation source: fine-focus sealed tube | 1491 reflections with $I > 2\sigma(I)$ |
| graphite | $R_{int} = 0.022$ |
| Rotation method data acquisition using $\omega$ and $\varphi$ scans. | $\theta_{max} = 26.4°$, $\theta_{min} = 2.7°$ |
| Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2009) | $h = -11 \rightarrow 11$ |
| $T_{min} = 0.098$, $T_{max} = 0.120$ | $k = -16 \rightarrow 15$ |
| 4524 measured reflections | $l = -11 \rightarrow 6$ |

*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 |
| $R[F^2 > 2\sigma(F^2)] = 0.027$ | $w = 1/[\sigma^2(F_o^2) + (0.0357P)^2 + 1.2795P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.066$ | $(\Delta/\sigma)_{max} = 0.004$ |
| $S = 1.07$ | $\Delta\rho_{max} = 0.77$ e Å$^{-3}$ |
| 1645 reflections | $\Delta\rho_{min} = -0.56$ e Å$^{-3}$ |

| | |
|---|---|
| 131 parameters | Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| 2 restraints | Extinction coefficient: 0.0072 (5) |
| Primary atom site location: structure-invariant direct methods | Absolute structure: Flack (1983), 416 Friedel pairs |
| Secondary atom site location: difference Fourier map | Flack 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 ($Å^2$)*

| | *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 ($Å^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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, °)*

| | | | |
|---|---|---|---|
| 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, °)*

| *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) |

**Fig. 1**

**Fig. 2**