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2,2,2-Tribromo-*N*-phenylacetamide

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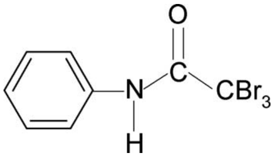
Key indicators: single-crystal X-ray study; *T* = 299 K; mean σ(C–C) = 0.029 Å; *R* factor = 0.079; *wR* factor = 0.237; data-to-parameter ratio = 11.0.

In the title compound, $C_8H_6Br_3NO$, the N—H bond is *anti* to the carbonyl bond in the side chain. The N—H hydrogen atom is involved in a two-centered bond as it shows simultaneous N—H···Br intra- and N—H···O intermolecular interactions in the structure. In the crystal, molecules are packed into column-like chains along the *b* axis through the N—H···O hydrogen bonds.

Related literature

For the preparation of the compound, see: Gowda *et al.* (2003). For related structures, see: Brown *et al.* (1966); Dou *et al.* (1994); Gowda *et al.* (2007, 2009).



Experimental

Crystal data

$C_8H_6Br_3NO$
M_r = 371.87
Orthorhombic, $Pca2_1$
a = 10.1863 (8) Å
b = 9.1483 (7) Å
c = 11.8856 (9) Å
V = 1107.59 (15) Å^3
Z = 4
Cu *Kα* radiation
μ = 13.22 mm^{-1}
T = 299 K
0.50 × 0.18 × 0.13 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.037, T_{max} = 0.178
2653 measured reflections
1311 independent reflections
1237 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
3 standard reflections
frequency: 120 min
intensity decay: 1.5%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.079
$wR(F^2)$ = 0.237
S = 1.05
1311 reflections
119 parameters
25 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.86 e Å^{-3}
$\Delta\rho_{min}$ = −1.18 e Å^{-3}
Absolute structure: Flack (1983), 276 Friedel pairs
Flack parameter: 0.00 (13)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^i	0.86	2.19	2.967 (13)	150
N1—H1*N*···Br1	0.86	2.68	3.123 (13)	114

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

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: FL2260).

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supporting information

Acta Cryst. (2009). E**65**, o2226 [doi:10.1107/S160053680903298X]

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S1. Comment

The structure of (I) has been determined (Fig. 1) as part of a study on the effect of ring and side chain substituents on the structures of *N*-aromatic amides (Dou *et al.*, 1994; Gowda *et al.*, 2007, 2009). The N—H bond in (I) is *anti* to the C=O bond in the side chain, similar to that observed in *N*-(phenyl)acetamide (Brown, 1966), 2,2,2-trichloro-*N*-(phenyl)-acetamide (Dou *et al.*, 1994), 2,2,2-trimethyl-*N*-(phenyl)acetamide (Gowda *et al.*, 2007) and other amides (Gowda *et al.*, 2009). The N—H hydrogen atom is involved as the donor in a two-centered bond; an intramolecular N—H···Br bond and an intermolecular N—H···O bond (Table 1). The N1—H1N···O1 bonds involved in the formation of molecular chains in the direction of the *b*-axis are shown Fig. 2.

S2. Experimental

The title compound was prepared from aniline, 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 from petroleum ether at room temperature.

S3. Refinement

The H atoms were positioned with idealized geometry using a riding model [N—H = 0.86 Å, 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).

The U^{ij} components of C2, C3, C4 and C5 were restrained to approximate isotropic behavoir.

The residual electron-density features are located in the region of Br3 and Br1. The highest peak is 1.25 Å from Br3 and the deepest hole is 0.75 Å from Br1.

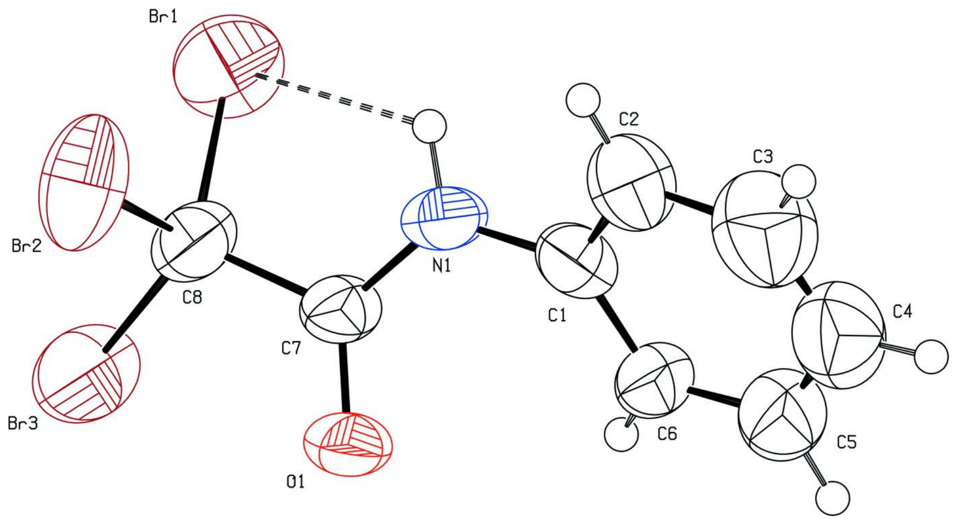

Figure 1

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.

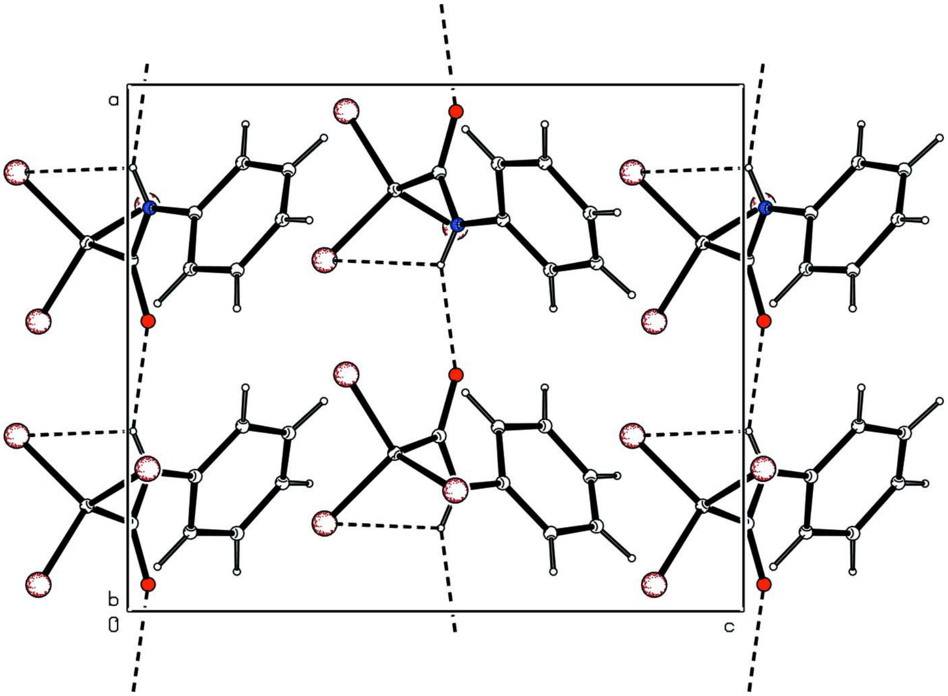

Figure 2

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

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Crystal data

$C_8H_6Br_3NO$
$M_r = 371.87$
Orthorhombic, $Pca2_1$
Hall symbol: P 2c -2ac

$a = 10.1863 (8)$ Å
$b = 9.1483 (7)$ Å
$c = 11.8856 (9)$ Å
$V = 1107.59 (15)$ Å^3

$Z = 4$
$F(000) = 696$
$D_x = 2.230$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54180$ Å
Cell parameters from 25 reflections
$\theta = 4.8$–20.7°
$\mu = 13.22$ mm^{-1}
$T = 299$ K
Needle, colourless
0.50 × 0.18 × 0.13 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.037$, $T_{\max} = 0.178$
2653 measured reflections
1311 independent reflections
1237 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.052$
$\theta_{\max} = 67.0°$, $\theta_{\min} = 4.8°$
$h = -12 \rightarrow 0$
$k = -10 \rightarrow 10$
$l = -11 \rightarrow 14$
3 standard reflections every 120 min
intensity decay: 1.5%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.079$
$wR(F^2) = 0.237$
$S = 1.05$
1311 reflections
119 parameters
25 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
$w = 1/[\sigma^2(F_o^2) + (0.1659P)^2 + 2.9656P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 1.86$ e Å^{-3}
$\Delta\rho_{\min} = -1.18$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0035 (8)
Absolute structure: Flack (1983), 276 Friedel pairs
Absolute structure parameter: 0.00 (13)

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 $F^2 > \sigma(F^2)$ 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	$U_{\text{iso}}*/U_{\text{eq}}$
C1	0.2439 (16)	0.3139 (13)	0.6091 (14)	0.057 (3)
C2	0.1483 (19)	0.2891 (18)	0.6869 (19)	0.078 (4)
H2	0.0753	0.3500	0.6914	0.094*
C3	0.162 (3)	0.169 (3)	0.761 (3)	0.110 (7)
H3	0.1010	0.1557	0.8185	0.132*
C4	0.256 (3)	0.077 (2)	0.751 (2)	0.095 (5)
H4	0.2575	−0.0070	0.7947	0.114*

C5	0.3521 (19)	0.1043 (17)	0.6774 (19)	0.079 (4)
H5	0.4247	0.0427	0.6773	0.095*
C6	0.3495 (16)	0.2176 (13)	0.6019 (17)	0.065 (4)
H6	0.4153	0.2297	0.5484	0.078*
C7	0.3328 (12)	0.5181 (14)	0.5056 (12)	0.053 (3)
C8	0.3002 (15)	0.6556 (17)	0.4339 (16)	0.069 (4)
N1	0.2341 (10)	0.4331 (11)	0.5359 (12)	0.058 (2)
H1N	0.1579	0.4526	0.5085	0.069*
O1	0.4489 (8)	0.5008 (11)	0.5332 (12)	0.076 (4)
Br1	0.1668 (3)	0.6121 (3)	0.3197 (2)	0.0988 (9)
Br2	0.2294 (3)	0.80125 (17)	0.5320 (2)	0.1007 (10)
Br3	0.4497 (2)	0.7263 (3)	0.3550 (3)	0.1230 (14)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.049 (6)	0.064 (7)	0.059 (8)	−0.012 (6)	−0.003 (6)	0.003 (6)
C2	0.075 (8)	0.080 (7)	0.079 (8)	−0.011 (6)	0.014 (7)	0.021 (6)
C3	0.112 (11)	0.110 (9)	0.108 (11)	−0.016 (8)	0.014 (9)	0.020 (8)
C4	0.106 (9)	0.085 (7)	0.095 (10)	−0.013 (8)	−0.013 (8)	0.014 (7)
C5	0.082 (8)	0.069 (6)	0.087 (9)	−0.002 (6)	−0.016 (7)	0.004 (6)
C6	0.060 (8)	0.052 (6)	0.083 (10)	0.004 (5)	−0.022 (7)	0.001 (6)
C7	0.038 (5)	0.066 (6)	0.054 (7)	0.001 (4)	0.005 (5)	0.017 (6)
C8	0.057 (8)	0.068 (7)	0.081 (11)	0.014 (6)	0.006 (7)	0.020 (7)
N1	0.037 (5)	0.068 (5)	0.068 (7)	0.005 (4)	−0.007 (5)	0.008 (6)
O1	0.035 (4)	0.085 (6)	0.109 (10)	−0.008 (4)	−0.007 (5)	0.042 (7)
Br1	0.1101 (17)	0.1157 (14)	0.0705 (12)	−0.0051 (11)	−0.0328 (12)	0.0179 (10)
Br2	0.164 (2)	0.0664 (9)	0.0719 (12)	0.0155 (10)	0.0171 (14)	0.0000 (8)
Br3	0.0680 (11)	0.1321 (19)	0.169 (3)	0.0095 (10)	0.0356 (14)	0.091 (2)

Geometric parameters (Å, º)

C1—C2	1.36 (3)	C5—H5	0.9300
C1—C6	1.39 (2)	C6—H6	0.9300
C1—N1	1.399 (19)	C7—O1	1.237 (16)
C2—C3	1.42 (3)	C7—N1	1.321 (16)
C2—H2	0.9300	C7—C8	1.555 (18)
C3—C4	1.27 (4)	C8—Br3	1.902 (16)
C3—H3	0.9300	C8—Br2	1.913 (17)
C4—C5	1.34 (4)	C8—Br1	1.960 (19)
C4—H4	0.9300	N1—H1N	0.8600
C5—C6	1.37 (2)		
C2—C1—C6	119.3 (15)	C5—C6—C1	116.9 (18)
C2—C1—N1	120.1 (15)	C5—C6—H6	121.6
C6—C1—N1	120.6 (15)	C1—C6—H6	121.6
C1—C2—C3	118.7 (19)	O1—C7—N1	125.5 (11)
C1—C2—H2	120.6	O1—C7—C8	117.0 (11)

C3—C2—H2	120.6	N1—C7—C8	117.5 (11)
C4—C3—C2	122 (3)	C7—C8—Br3	111.9 (9)
C4—C3—H3	119.2	C7—C8—Br2	108.1 (11)
C2—C3—H3	119.2	Br3—C8—Br2	111.4 (9)
C3—C4—C5	119 (2)	C7—C8—Br1	111.3 (11)
C3—C4—H4	120.3	Br3—C8—Br1	106.4 (9)
C5—C4—H4	120.3	Br2—C8—Br1	107.6 (7)
C4—C5—C6	123.6 (18)	C7—N1—C1	125.0 (11)
C4—C5—H5	118.2	C7—N1—H1N	117.5
C6—C5—H5	118.2	C1—N1—H1N	117.5

C6—C1—C2—C3	2 (3)	N1—C7—C8—Br3	−159.7 (12)
N1—C1—C2—C3	−179.1 (19)	O1—C7—C8—Br2	−100.0 (14)
C1—C2—C3—C4	−5 (4)	N1—C7—C8—Br2	77.2 (16)
C2—C3—C4—C5	8 (4)	O1—C7—C8—Br1	142.1 (13)
C3—C4—C5—C6	−7 (4)	N1—C7—C8—Br1	−40.7 (17)
C4—C5—C6—C1	4 (3)	O1—C7—N1—C1	4 (3)
C2—C1—C6—C5	−2 (2)	C8—C7—N1—C1	−173.4 (14)
N1—C1—C6—C5	179.6 (15)	C2—C1—N1—C7	139.6 (18)
O1—C7—C8—Br3	23.1 (19)	C6—C1—N1—C7	−42 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···O1^{i}	0.86	2.19	2.967 (13)	150
N1—H1*N*···Br1	0.86	2.68	3.123 (13)	114

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