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2-(4-Bromophenyl)acetohydrazide

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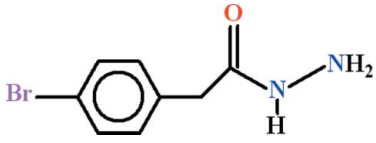
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.023; wR factor = 0.059; data-to-parameter ratio = 15.8.

In the title compound, $C_8H_9BrN_2O$, the 1-bromo-4-methylbenzene group and the formic hydrazide moiety [r.m.s. deviations of 0.0129 and 0.0038 Å] are oriented at a dihedral angle of 80.66 (11)°. In the crystal, molecules are linked *via* strong N—H···O hydrogen bonds, leading to the formation of chains in the [010] direction. These chains are linked *via* weaker N—H···N and N—H···O hydrogen bonds, with $R_2^2(7)$ and $R_3^2(7)$ ring motifs, forming a two-dimensional network parallel to (001).

Related literature

For background literature and the crystal structure of 2-chlorobenzohydrazide, see: Ahmad *et al.* (2012). For graph-set notation, see: Bernstein *et al.* (1995).



Experimental

Crystal data

$C_8H_9BrN_2O$
M_r = 229.07
Monoclinic, $P2_1$
a = 6.0798 (2) Å
b = 4.8565 (1) Å
c = 15.1126 (5) Å
β = 98.003 (2)°
V = 441.88 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 4.60 mm^{-1}
T = 296 K
0.36 × 0.23 × 0.22 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.298, T_{max} = 0.366
4331 measured reflections
1814 independent reflections
1677 reflections with $I > 2\sigma(I)$
R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.059
S = 1.06
1814 reflections
115 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}
Absolute structure: Flack (1983), 583 Friedel pairs
Flack parameter: 0.007 (11)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	2.02	2.863 (3)	165
N2—H2A···N2^{ii}	0.84 (4)	2.37 (4)	3.192 (4)	167 (3)
N2—H2B···O1^{iii}	0.73 (3)	2.59 (4)	3.230 (3)	147 (4)

Symmetry codes: (i) $x, y+1, z$; (ii) $-x-1, y-\frac{1}{2}, -z+1$; (iii) $-x, y+\frac{1}{2}, -z+1$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2429).

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supplementary materials

Acta Cryst. (2012). E**68**, o2269 [doi:10.1107/S1600536812027006]

2-(4-Bromophenyl)acetohydrazide

Shakeel Ahmad, Abdul Jabbar, Muhammad Tahir Hussain and M. Nawaz Tahir

Comment

Recently, we have reported the crystal structure of 2-chlorobenzohydrazide (Ahmad *et al.*, 2012). In continuation of this work we have synthesized the title compound, a hydrazide derivative, and report herein on its crystal structure.

In the title molecule, Fig. 1, the 1-bromo-4-methylbenzene group A (C1–C7/Br1) and the formic hydrazide moiety B (O1/C8/N1/N2) are planar with r. m. s. deviations of 0.0129 Å and 0.0038 Å, respectively. The dihedral angle between these mean planes, A/B, is 80.66 (11)°.

In the crystal, molecules are linked via N—H···O hydrogen bonds to form one-dimensional polymeric chains along [010]. These chains are linked via N-H···N and N-H..O hydrogen bonds to form a two-dimensional polymeric network in (001). The hydrogen bonds give rise to $R_2^2(7)$ and $R_3^2(7)$ ring motifs (Bernstein *et al.*, 1995; Table 1 and Fig. 2).

Experimental

2-(4-Bromophenyl)acetic acid (4.42 g, 0.022 mol) was converted to methyl 2-(4-bromophenyl)acetate by refluxing in methanol (25 ml) in the presence of catalytic amount of sulfuric acid. This ester was then converted into the title compound by refluxing with hydrazine hydrate (80%, 10 ml) in dry methanol. The title compound was purified by recrystallization from dry methanol, giving colourless rod-like crystals [M.p. 438–439 K].

Refinement

The coordinates of the H-atoms of the NH_2 group were refined with $U_{iso}(H) = 1.2U_{eq}(N)$. The remainder of the H-atoms were included in calculated positions and treated as riding atoms: N–H = 0.86 Å, C–H = 0.93–0.97 Å, with $U_{iso}(H) = 1.2U_{eq}(N,C)$.

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).

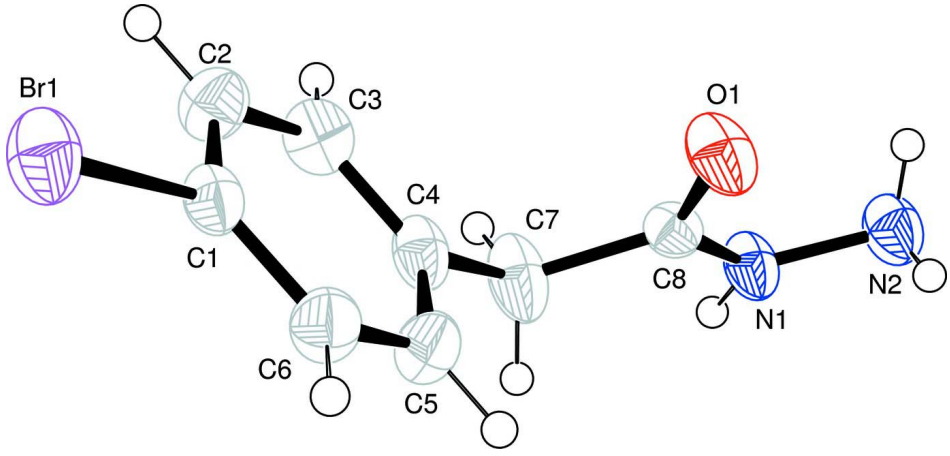

Figure 1

View of the title molecule with the atom numbering. The displacement ellipsoids are drawn at the 50% probability level.

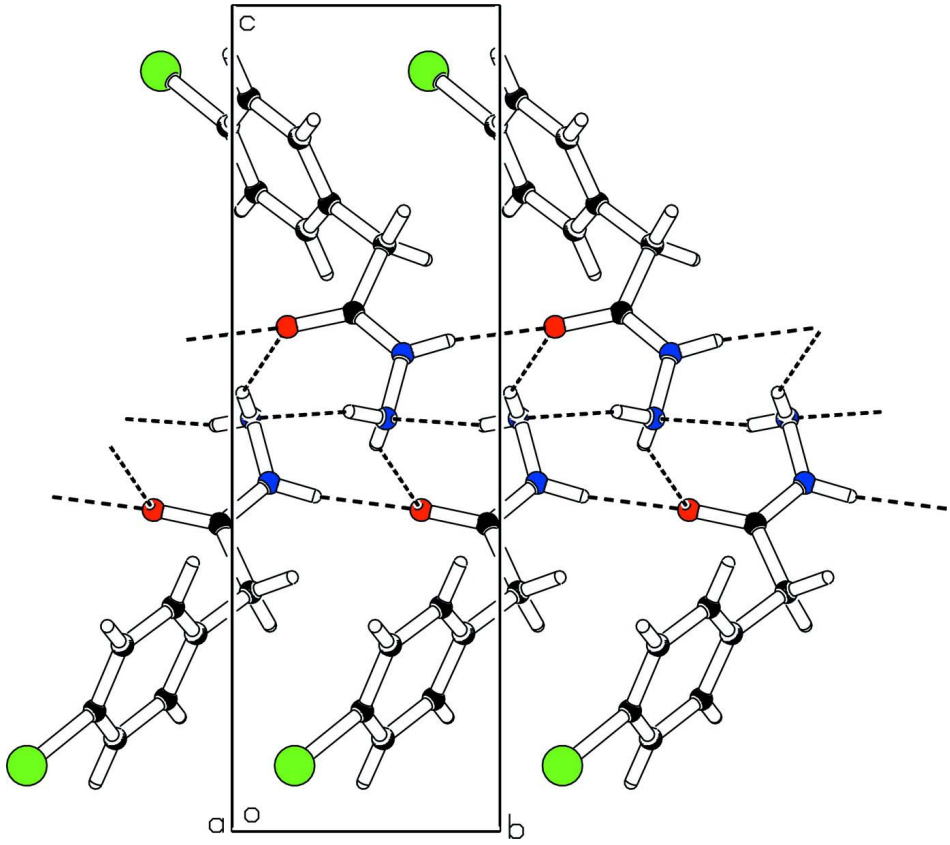

Figure 2

A view along the a axis of the crystal packing of the title compound. The two-dimensional hydrogen bonded network extends in the plane (001). Hydrogen bonds are shown as dashed lines - see Table 1 for details.

2-(4-Bromophenyl)acetohydrazide

Crystal data

$C_8H_9BrN_2O$
$M_r = 229.07$
Monoclinic, $P2_1$
Hall symbol: P 2yb
$a = 6.0798$ (2) Å
$b = 4.8565$ (1) Å
$c = 15.1126$ (5) Å
$\beta = 98.003$ (2)°
$V = 441.88$ (2) Å^3
$Z = 2$
$F(000) = 228$
$D_x = 1.722$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1677 reflections
$\theta = 2.7$–$28.3°$
$\mu = 4.60$ mm^{-1}
$T = 296$ K
Rod, colourless
0.36 × 0.23 × 0.22 mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.50 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
$T_{min} = 0.298$, $T_{max} = 0.366$
4331 measured reflections
1814 independent reflections
1677 reflections with $I > 2\sigma(I)$
$R_{int} = 0.023$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.7°$
$h = -8 \rightarrow 8$
$k = -6 \rightarrow 4$
$l = -20 \rightarrow 20$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.059$
$S = 1.06$
1814 reflections
115 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.024P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}
Absolute structure: Flack (1983), 583 Friedel pairs
Flack parameter: 0.007 (11)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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	0.42290 (4)	0.23371 (7)	0.07962 (1)	0.0435 (1)
O1	−0.1520 (3)	0.7065 (5)	0.38909 (13)	0.0457 (6)
N1	−0.2370 (4)	1.1385 (4)	0.42196 (15)	0.0364 (7)

N2	−0.3296 (5)	1.0741 (6)	0.49999 (17)	0.0420 (8)
C1	0.2739 (4)	0.4860 (5)	0.14603 (16)	0.0315 (8)
C2	0.0587 (5)	0.5632 (6)	0.11262 (19)	0.0395 (9)
C3	−0.0499 (4)	0.7519 (8)	0.16041 (16)	0.0407 (8)
C4	0.0518 (5)	0.8638 (5)	0.23979 (17)	0.0362 (8)
C5	0.2669 (5)	0.7798 (7)	0.27159 (17)	0.0416 (12)
C6	0.3788 (5)	0.5900 (6)	0.22578 (17)	0.0381 (8)
C7	−0.0693 (6)	1.0694 (6)	0.2896 (2)	0.0494 (10)
C8	−0.1528 (4)	0.9529 (5)	0.37211 (16)	0.0289 (7)
H1	−0.23523	1.30826	0.40587	0.0437*
H2	−0.01156	0.49007	0.05918	0.0474*
H2A	−0.416 (5)	0.940 (8)	0.491 (2)	0.0503*
H2B	−0.230 (6)	1.040 (8)	0.531 (2)	0.0503*
H3	−0.19442	0.80410	0.13848	0.0488*
H5	0.33752	0.85271	0.32499	0.0500*
H6	0.52191	0.53407	0.24842	0.0457*
H7A	−0.19487	1.14063	0.24952	0.0591*
H7B	0.02941	1.22250	0.30749	0.0591*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0503 (2)	0.0403 (2)	0.0433 (1)	0.0101 (2)	0.0184 (1)	−0.0001 (2)
O1	0.0663 (11)	0.0214 (11)	0.0547 (10)	0.0035 (12)	0.0269 (9)	0.0049 (11)
N1	0.0533 (14)	0.0226 (11)	0.0367 (11)	0.0007 (9)	0.0184 (10)	0.0019 (8)
N2	0.0511 (16)	0.0378 (14)	0.0407 (14)	0.0000 (12)	0.0193 (12)	−0.0020 (11)
C1	0.0367 (13)	0.0284 (14)	0.0315 (12)	0.0012 (10)	0.0123 (10)	0.0007 (10)
C2	0.0393 (14)	0.0426 (17)	0.0356 (13)	0.0034 (12)	0.0021 (12)	−0.0016 (12)
C3	0.0373 (11)	0.0407 (16)	0.0442 (12)	0.0084 (16)	0.0063 (10)	0.0062 (17)
C4	0.0523 (16)	0.0247 (13)	0.0352 (13)	0.0029 (11)	0.0185 (12)	0.0034 (10)
C5	0.0488 (14)	0.044 (3)	0.0321 (11)	−0.0038 (14)	0.0061 (11)	−0.0051 (12)
C6	0.0344 (14)	0.0440 (16)	0.0355 (13)	0.0028 (12)	0.0040 (12)	0.0009 (12)
C7	0.077 (2)	0.0298 (16)	0.0479 (17)	0.0083 (15)	0.0319 (16)	0.0057 (13)
C8	0.0312 (12)	0.0221 (13)	0.0338 (12)	0.0001 (10)	0.0064 (10)	−0.0008 (9)

Geometric parameters (Å, º)

Br1—C1	1.893 (2)	C4—C5	1.390 (4)
O1—C8	1.224 (3)	C4—C7	1.503 (4)
N1—N2	1.411 (4)	C5—C6	1.387 (4)
N1—C8	1.323 (3)	C7—C8	1.520 (4)
N1—H1	0.8600	C2—H2	0.9300
N2—H2B	0.73 (3)	C3—H3	0.9300
N2—H2A	0.84 (4)	C5—H5	0.9300
C1—C2	1.387 (4)	C6—H6	0.9300
C1—C6	1.379 (4)	C7—H7A	0.9700
C2—C3	1.389 (4)	C7—H7B	0.9700
C3—C4	1.382 (4)		
N2—N1—C8	123.8 (2)	O1—C8—C7	123.0 (2)

C8—N1—H1	118.00	N1—C8—C7	114.4 (2)
N2—N1—H1	118.00	O1—C8—N1	122.5 (2)
N1—N2—H2B	101 (3)	C1—C2—H2	121.00
H2A—N2—H2B	112 (4)	C3—C2—H2	121.00
N1—N2—H2A	111 (2)	C2—C3—H3	119.00
Br1—C1—C2	118.61 (19)	C4—C3—H3	119.00
Br1—C1—C6	120.2 (2)	C4—C5—H5	119.00
C2—C1—C6	121.2 (2)	C6—C5—H5	119.00
C1—C2—C3	118.8 (2)	C1—C6—H6	121.00
C2—C3—C4	121.5 (3)	C5—C6—H6	121.00
C3—C4—C7	120.3 (3)	C4—C7—H7A	109.00
C3—C4—C5	118.1 (3)	C4—C7—H7B	109.00
C5—C4—C7	121.6 (3)	C8—C7—H7A	109.00
C4—C5—C6	121.7 (3)	C8—C7—H7B	109.00
C1—C6—C5	118.7 (3)	H7A—C7—H7B	108.00
C4—C7—C8	114.0 (2)		

N2—N1—C8—O1	1.3 (4)	C2—C3—C4—C7	179.5 (3)
N2—N1—C8—C7	178.1 (3)	C3—C4—C5—C6	0.1 (4)
Br1—C1—C2—C3	−178.9 (2)	C7—C4—C5—C6	179.9 (3)
C6—C1—C2—C3	1.0 (4)	C3—C4—C7—C8	104.7 (3)
Br1—C1—C6—C5	178.4 (2)	C5—C4—C7—C8	−75.1 (4)
C2—C1—C6—C5	−1.5 (4)	C4—C5—C6—C1	1.0 (4)
C1—C2—C3—C4	0.2 (5)	C4—C7—C8—O1	−10.6 (4)
C2—C3—C4—C5	−0.7 (4)	C4—C7—C8—N1	172.7 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.02	2.863 (3)	165
N2—H2*A*···N2^{ii}	0.84 (4)	2.37 (4)	3.192 (4)	167 (3)
N2—H2*B*···O1^{iii}	0.73 (3)	2.59 (4)	3.230 (3)	147 (4)

Symmetry codes: (i) *x*, *y*+1, *z*; (ii) −*x*−1, *y*−1/2, −*z*+1; (iii) −*x*, *y*+1/2, −*z*+1.