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3-Bromo-4-methoxybenzaldehyde 4-nitrophenylhydrazone

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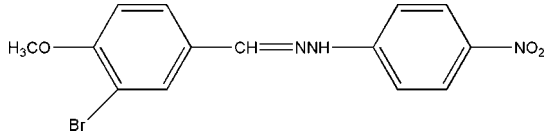
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.091; wR factor = 0.176; data-to-parameter ratio = 15.6.

In the title compound, $C_{14}H_{12}BrN_3O_3$, the two benzene rings are slightly twisted, making a dihedral angle of 9.9 (3)°. The crystal packing is stabilized by weak intermolecular N—H···O and C—H···O hydrogen bonds.

Related literature

For related literature, see: Okabe *et al.* (1993).



Experimental

Crystal data

$C_{14}H_{12}BrN_3O_3$
M_r = 350.18
Orthorhombic, *Pccn*
a = 7.2800 (15) Å
b = 14.678 (3) Å
c = 27.368 (6) Å
V = 2924.4 (10) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 2.82 mm^{-1}
T = 298 (2) K
0.27 × 0.25 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.486, T_{max} = 0.548
30041 measured reflections
2980 independent reflections
2456 reflections with $I > 2\sigma(I)$
R_{int} = 0.060

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.091
$wR(F^2)$ = 0.176
S = 1.22
2980 reflections
191 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.93 e Å^{-3}
$\Delta\rho_{min}$ = −0.58 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O2^{i}	0.86	2.19	3.035 (6)	166
C7—H7···O1^{i}	0.93	2.48	3.400 (8)	172

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN3064).

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supplementary materials

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3-Bromo-4-methoxybenzaldehyde 4-nitrophenylhydrazone

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Comment

4-Nitrophenylhydrazine has applications in organic synthesis and some of its derivatives have been shown to be potentially DNA-damaging and mutagenic agents (Okabe *et al.*,1993). As a continuation of this work, we report the synthesis and crystal structure of the title compound(I).

The 4-nitrophenyl group and the methyl benzene rings areslightly twisted making a dihedral angle of 9.9 (3)° (Fig. 1). The N1/O1/O2 nitro group is co-planar with its attached benzene ring [dihedral angle =1.4 (2)°].

The intermolecular N—H···O and C—H···O hydrogen bonds can help stabilizing the molecular structure·(Table 1, Fig.2)

Experimental

4-Nitrophenylhydrazine (1 mmol, 0.153 g) was dissolved in anhydrous methanol, H_2SO_4 (98% 0.5 ml) was added to this, the mixture was stirred for several minitutes at 351 K, 3-Bromo-4-methoxybenzaldehyde (1 mmol 0.215 g) in methanol (8 ml) was added dropwise and the mixture was stirred at refluxing temperature for 2 h. The product was isolated and recrystallized in dichloromethane, brown single crystals of (I) was obtained after 1 d.

Refinement

All H atoms attached to C atoms and N atom were fixed geometrically and treated as riding with C—H = 0.93Å (aromatic) or 0.96Å (methy) and N—H = 0.86Å with U_{iso}(H) = 1.2U_{eq}($C_{aromatic}$, N) or U_{iso}(H) = 1.5U_{eq}(methyl).

Figures

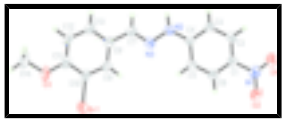

Fig. 1. Molecular view of (I) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

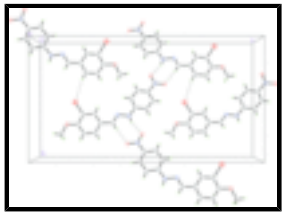

Fig. 2. Partial packing view of (I), showing the intermolecular hydrogen bonds as dashed lines.

3-Bromo-4-methoxybenzaldehyde 4-nitrophenylhydrazone

Crystal data

$C_{14}H_{12}BrN_3O_3$	F_{000} = 1408
M_r = 350.18	D_x = 1.591 Mg m^{-3}
Orthorhombic, *Pccn*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ab 2ac	Cell parameters from 1248 reflections
a = 7.2800 (15) Å	θ = 2.5–27.8º
b = 14.678 (3) Å	μ = 2.82 mm^{-1}
c = 27.368 (6) Å	*T* = 298 (2) K
V = 2924.4 (10) Å^3	Block, brown
Z = 8	0.27 × 0.25 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2980 independent reflections
Radiation source: fine-focus sealed tube	2456 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.060
T = 298(2) K	θ_{max} = 26.5º
ω scans	θ_{min} = 2.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −9→8
T_{min} = 0.486, T_{max} = 0.548	*k* = −18→18
30041 measured reflections	*l* = −34→34

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.091	H-atom parameters constrained
$wR(F^2)$ = 0.176	$w = 1/[\sigma^2(F_o^2) + (0.045P)^2 + 6.3175P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.22	$(\Delta/\sigma)_{max} < 0.001$
2980 reflections	$\Delta\rho_{max}$ = 0.93 e Å^{-3}
191 parameters	$\Delta\rho_{min}$ = −0.58 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.11817 (15)	0.97770 (5)	0.17684 (2)	0.1058 (4)
O1	0.3606 (7)	1.1359 (3)	0.58357 (17)	0.0930 (14)
O2	0.4495 (6)	1.2106 (3)	0.51958 (19)	0.0857 (13)
O3	0.0204 (7)	0.7930 (3)	0.14235 (15)	0.0887 (14)
N1	0.3772 (7)	1.1431 (3)	0.5392 (2)	0.0687 (13)
N2	0.1160 (7)	0.8638 (3)	0.42050 (15)	0.0642 (12)
H2	0.0786	0.8142	0.4340	0.077*
N3	0.1076 (6)	0.8735 (3)	0.37049 (16)	0.0630 (12)
C1	0.3092 (7)	1.0709 (4)	0.50809 (19)	0.0556 (13)
C2	0.2439 (8)	0.9916 (4)	0.5293 (2)	0.0602 (13)
H2A	0.2421	0.9849	0.5630	0.072*
C3	0.1816 (7)	0.9227 (4)	0.49932 (18)	0.0573 (13)
H3	0.1378	0.8690	0.5131	0.069*
C4	0.1836 (7)	0.9327 (3)	0.44878 (18)	0.0509 (12)
C5	0.2542 (8)	1.0125 (4)	0.42843 (19)	0.0591 (13)
H5	0.2577	1.0196	0.3947	0.071*
C6	0.3184 (8)	1.0802 (4)	0.4581 (2)	0.0615 (14)
H6	0.3683	1.1328	0.4445	0.074*
C7	0.0422 (8)	0.8060 (4)	0.3473 (2)	0.0666 (15)
H7	0.0015	0.7557	0.3648	0.080*
C8	0.0284 (8)	0.8046 (4)	0.2942 (2)	0.0622 (14)
C9	0.0654 (8)	0.8806 (4)	0.2657 (2)	0.0621 (14)
H9	0.0943	0.9358	0.2805	0.075*
C10	0.0594 (8)	0.8745 (4)	0.2153 (2)	0.0648 (15)
C11	0.0189 (8)	0.7920 (4)	0.1923 (2)	0.0656 (15)
C12	−0.0173 (9)	0.7173 (4)	0.2203 (3)	0.0802 (19)
H12	−0.0449	0.6620	0.2055	0.096*
C13	−0.0130 (9)	0.7237 (4)	0.2705 (2)	0.0745 (17)
H13	−0.0388	0.6722	0.2891	0.089*
C14	−0.0039 (13)	0.7075 (5)	0.1180 (3)	0.112 (3)
H14A	0.0900	0.6658	0.1284	0.167*
H14B	0.0044	0.7163	0.0833	0.167*
H14C	−0.1224	0.6830	0.1260	0.167*

Atomic displacement parameters (Å^2)

U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

Br1	0.1924 (10)	0.0635 (4)	0.0616 (4)	0.0057 (5)	−0.0070 (5)	−0.0025 (3)
O1	0.119 (4)	0.090 (3)	0.069 (3)	−0.005 (3)	−0.008 (3)	−0.017 (2)
O2	0.091 (3)	0.057 (2)	0.108 (3)	−0.012 (2)	−0.009 (3)	−0.005 (2)
O3	0.126 (4)	0.075 (3)	0.064 (2)	0.023 (3)	−0.029 (3)	−0.030 (2)
N1	0.063 (3)	0.061 (3)	0.082 (4)	0.008 (3)	−0.007 (3)	−0.009 (3)
N2	0.082 (3)	0.059 (3)	0.052 (2)	−0.010 (3)	−0.002 (2)	0.008 (2)
N3	0.071 (3)	0.067 (3)	0.051 (2)	−0.003 (2)	−0.003 (2)	0.002 (2)
C1	0.053 (3)	0.052 (3)	0.062 (3)	0.006 (2)	−0.011 (3)	−0.002 (2)
C2	0.063 (3)	0.066 (3)	0.052 (3)	0.009 (3)	−0.001 (3)	0.006 (3)
C3	0.062 (3)	0.054 (3)	0.055 (3)	0.000 (3)	−0.003 (3)	0.009 (2)
C4	0.045 (3)	0.053 (3)	0.055 (3)	0.002 (2)	−0.003 (2)	0.002 (2)
C5	0.066 (3)	0.060 (3)	0.050 (3)	−0.009 (3)	−0.003 (3)	0.009 (2)
C6	0.062 (3)	0.050 (3)	0.072 (4)	−0.003 (3)	−0.004 (3)	0.014 (3)
C7	0.071 (4)	0.057 (3)	0.071 (4)	−0.006 (3)	−0.010 (3)	−0.002 (3)
C8	0.056 (3)	0.065 (4)	0.065 (3)	−0.004 (3)	−0.007 (3)	−0.010 (3)
C9	0.075 (4)	0.053 (3)	0.059 (3)	0.006 (3)	−0.015 (3)	−0.012 (3)
C10	0.083 (4)	0.052 (3)	0.060 (3)	0.016 (3)	−0.013 (3)	−0.012 (3)
C11	0.065 (4)	0.066 (4)	0.066 (3)	0.014 (3)	−0.019 (3)	−0.019 (3)
C12	0.088 (5)	0.055 (3)	0.098 (5)	−0.005 (3)	−0.023 (4)	−0.028 (3)
C13	0.083 (4)	0.062 (4)	0.078 (4)	−0.010 (3)	−0.012 (3)	−0.004 (3)
C14	0.161 (8)	0.086 (5)	0.088 (5)	0.034 (5)	−0.040 (5)	−0.044 (4)

Geometric parameters (Å, °)

Br1—C10	1.893 (6)	C5—C6	1.366 (7)
O1—N1	1.226 (6)	C5—H5	0.9300
O2—N1	1.244 (6)	C6—H6	0.9300
O3—C11	1.367 (7)	C7—C8	1.456 (8)
O3—C14	1.432 (7)	C7—H7	0.9300
N1—C1	1.447 (7)	C8—C13	1.385 (8)
N2—C4	1.364 (6)	C8—C9	1.389 (8)
N2—N3	1.377 (6)	C9—C10	1.382 (7)
N2—H2	0.8600	C9—H9	0.9300
N3—C7	1.269 (7)	C10—C11	1.398 (7)
C1—C6	1.376 (7)	C11—C12	1.363 (9)
C1—C2	1.384 (7)	C12—C13	1.379 (9)
C2—C3	1.379 (7)	C12—H12	0.9300
C2—H2A	0.9300	C13—H13	0.9300
C3—C4	1.391 (7)	C14—H14A	0.9600
C3—H3	0.9300	C14—H14B	0.9600
C4—C5	1.396 (7)	C14—H14C	0.9600
C11—O3—C14	117.0 (5)	N3—C7—H7	118.8
O1—N1—O2	122.5 (5)	C8—C7—H7	118.8
O1—N1—C1	119.1 (5)	C13—C8—C9	117.8 (5)
O2—N1—C1	118.4 (5)	C13—C8—C7	119.6 (6)
C4—N2—N3	120.3 (4)	C9—C8—C7	122.4 (5)
C4—N2—H2	119.9	C10—C9—C8	120.2 (5)
N3—N2—H2	119.9	C10—C9—H9	119.9
C7—N3—N2	115.7 (5)	C8—C9—H9	119.9

C6—C1—C2	121.1 (5)	C9—C10—C11	120.8 (6)
C6—C1—N1	119.7 (5)	C9—C10—Br1	119.7 (4)
C2—C1—N1	119.2 (5)	C11—C10—Br1	119.4 (4)
C3—C2—C1	118.8 (5)	C12—C11—O3	125.0 (5)
C3—C2—H2A	120.6	C12—C11—C10	118.9 (5)
C1—C2—H2A	120.6	O3—C11—C10	116.1 (6)
C2—C3—C4	120.7 (5)	C11—C12—C13	120.1 (5)
C2—C3—H3	119.7	C11—C12—H12	119.9
C4—C3—H3	119.7	C13—C12—H12	119.9
N2—C4—C3	118.8 (5)	C12—C13—C8	122.0 (6)
N2—C4—C5	121.9 (5)	C12—C13—H13	119.0
C3—C4—C5	119.3 (5)	C8—C13—H13	119.0
C6—C5—C4	120.0 (5)	O3—C14—H14A	109.5
C6—C5—H5	120.0	O3—C14—H14B	109.5
C4—C5—H5	120.0	H14A—C14—H14B	109.5
C5—C6—C1	120.2 (5)	O3—C14—H14C	109.5
C5—C6—H6	119.9	H14A—C14—H14C	109.5
C1—C6—H6	119.9	H14B—C14—H14C	109.5
N3—C7—C8	122.5 (5)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O2^{i}	0.86	2.19	3.035 (6)	166
C7—H7···O1^{i}	0.93	2.48	3.400 (8)	172

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

Fig. 1

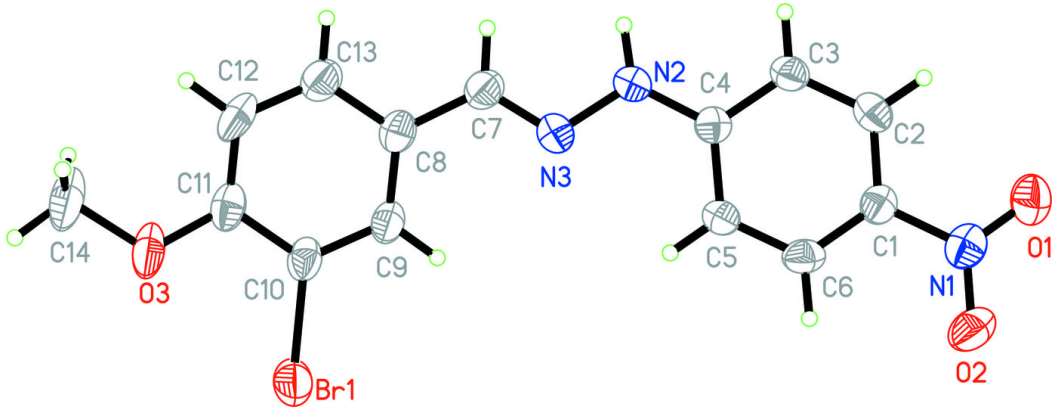

Fig. 2