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4-Bromo-1-nitrobenzene

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Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.006 Å; R factor = 0.045; wR factor = 0.119; data-to-parameter ratio = 15.7.

The non-H atoms of the title molecule, $C_6H_4BrNO_2$, are essentially coplanar with an r.m.s. deviation of 0.040 Å. In the crystal, π–π stacking occurs between parallel benzene rings of adjacent molecules with centroid–centroid distances of 3.643 (3) and 3.741 (3) Å. Weak intermolecular C—H···O hydrogen bonding and short Br···O contacts [3.227 (4) 3.401 (4) Å] are also observed in the crystal structure. The crystal studied was a non-morohedral twin with a 26.1 (6)% minor component.

Related literature

For the structure of 2-bromonitrobenzene, see: Fronczek (2006). For the structure of 3-bromonitrobenzene, see: Charlton & Trotter (1963).

Experimental

Crystal data

$C_6H_4BrNO_2$ M_r = 202.01
Triclinic, $P\overline{1}$
a = 6.3676 (6) Å
b = 7.3635 (7) Å
c = 7.6798 (7) Å
α = 65.554 (9)°
β = 87.705 (8)°
γ = 88.884 (8)°
V = 327.54 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 6.20 mm^{-1}
T = 100 K
0.20 × 0.10 × 0.05 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.414, T_{max} = 1.000
2142 measured reflections
1443 independent reflections
1365 reflections with $I > 2\sigma(I)$
R_{int} = 0.052

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.119
S = 1.07
1443 reflections
92 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.91 e Å^{-3}
$\Delta\rho_{min}$ = −1.58 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O1^{i}	0.95	2.52	3.359 (6)	147
C5—H5···O2^{ii}	0.95	2.54	3.276 (6)	135

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $-x+1, -y, -z+2$.

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

We thank the Higher Education Commission of Pakistan and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5150).

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supporting information

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4-Bromo-1-nitrobenzene

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S1. Comment

4-Bromo-1-nitrobenzene (Scheme I) was synthesized as a precursor that will be used in the synthesis of 4,4′-bis(amino-phenoxy)biphenyl (the compound is also commercially available: http://www.chemindustry.com/chemicals/815494.html). The molecule is flat (Fig. 1) as the nitro substituent is co-planar with the aromatic ring. π-π stacking occrs between parallel benzene rings of adjacent molecules, centroids distance between C1-ring and C1^{i}-ring (symmetry code: (i) 1-x, -5, 1-z) is 3.643 (3) Å and that between C1-ring and C1^{ii}-ring (symmetry code: (ii) 1-x, 1-y, 1-z) is 3.741 (3) Å. Intermolecular weak C—H···O hydrogen bonding (Table 1) and the short Br···O contacts [3.227 (4), 3.401 (4) Å] are observed in the crystal structure.

S2. Experimental

The nitrating mixture cosisted of 5 ml conc. HNO_3 and 5 ml conc. H_2SO_4 kept at 273 K. Bromobenzene (2.6 ml) was added. The temperature was then raised to about 333 K for 3 h. The mixture was added to water (200 ml); the organic compound was extracted by using dichloromethane. The solvent was dried and then alllowed to evaporate to yield the product in 70% yield.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C—H 0.95 Å, U_{iso}(H) 1.2U_{eq}(C)] and were included in the refinement in the riding model approximation.

The crystal is a non-merohedral twin; the separation of the two domains was effected by *CrysAlis PRO* (Agilent, 2010).

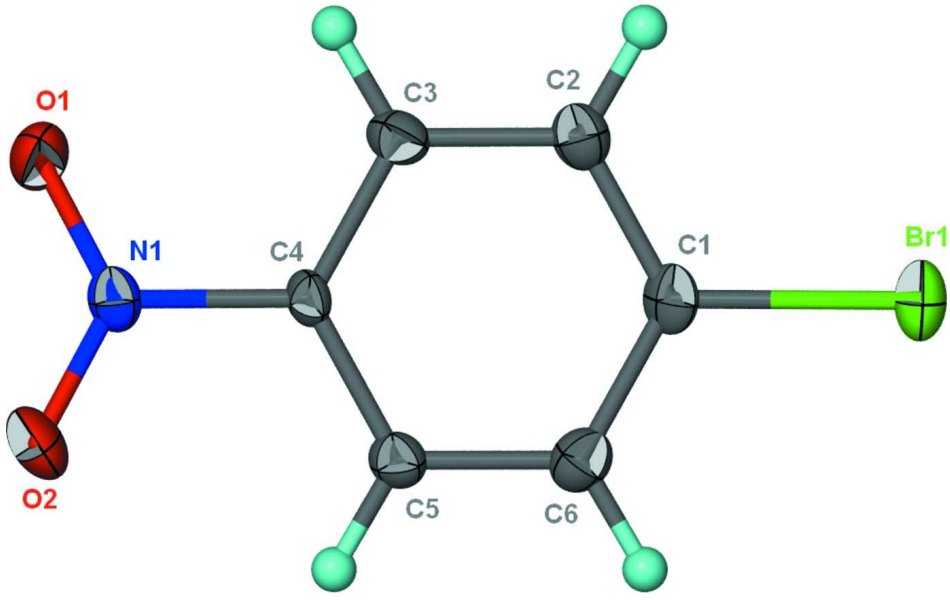

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_6H_4BrNO_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

4-Bromo-1-nitrobenzene

Crystal data

$C_6H_4BrNO_2$
$M_r = 202.01$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.3676\ (6)$ Å
$b = 7.3635\ (7)$ Å
$c = 7.6798\ (7)$ Å
$\alpha = 65.554\ (9)°$
$\beta = 87.705\ (8)°$
$\gamma = 88.884\ (8)°$
$V = 327.54\ (5)$ Å^3

$Z = 2$
$F(000) = 196$
$D_x = 2.048$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1590 reflections
$\theta = 2.9–28.3°$
$\mu = 6.20$ mm^{-1}
$T = 100$ K
Block, colorless
0.20 × 0.10 × 0.05 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)

$T_{min} = 0.414$, $T_{max} = 1.000$
2142 measured reflections
1443 independent reflections
1365 reflections with $I > 2\sigma(I)$
$R_{int} = 0.052$
$\theta_{max} = 27.5°$, $\theta_{min} = 2.9°$
$h = -8\rightarrow8$
$k = -9\rightarrow9$
$l = -9\rightarrow9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.119$
$S = 1.07$
1443 reflections
92 parameters
0 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
$w = 1/[\sigma^2(F_o^2) + (0.0671P)^2 + 0.4717P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.91$ e Å^{-3}
$\Delta\rho_{min} = -1.58$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.83556 (7)	0.24763 (7)	0.14671 (6)	0.01978 (19)
O1	0.0573 (5)	0.3167 (6)	0.7390 (5)	0.0238 (8)
O2	0.3071 (6)	0.2117 (6)	0.9389 (5)	0.0245 (8)
N1	0.2383 (6)	0.2623 (6)	0.7787 (5)	0.0151 (7)
C6	0.7151 (7)	0.1751 (7)	0.5288 (7)	0.0173 (9)
H6	0.8526	0.1212	0.5575	0.021*
C5	0.5810 (7)	0.1811 (7)	0.6722 (6)	0.0146 (9)
H5	0.6251	0.1326	0.8004	0.018*
C2	0.4465 (7)	0.3256 (7)	0.2969 (7)	0.0173 (9)
H2	0.4024	0.3750	0.1687	0.021*
C3	0.3121 (7)	0.3291 (7)	0.4408 (6)	0.0159 (9)
H3	0.1731	0.3792	0.4130	0.019*
C1	0.6478 (7)	0.2483 (6)	0.3429 (6)	0.0157 (9)
C4	0.3809 (7)	0.2594 (6)	0.6254 (6)	0.0125 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0204 (3)	0.0213 (3)	0.0206 (3)	−0.00244 (19)	0.00662 (18)	−0.0122 (2)
O1	0.0149 (16)	0.038 (2)	0.0232 (18)	0.0024 (15)	0.0001 (13)	−0.0174 (16)
O2	0.0299 (19)	0.032 (2)	0.0131 (16)	0.0028 (16)	−0.0003 (14)	−0.0107 (15)
N1	0.0173 (18)	0.0152 (18)	0.0156 (18)	−0.0013 (14)	0.0010 (14)	−0.0093 (15)
C6	0.015 (2)	0.017 (2)	0.021 (2)	−0.0008 (17)	0.0000 (17)	−0.0089 (19)
C5	0.016 (2)	0.015 (2)	0.014 (2)	0.0014 (16)	−0.0023 (16)	−0.0076 (17)
C2	0.020 (2)	0.018 (2)	0.016 (2)	−0.0004 (18)	0.0001 (17)	−0.0094 (18)
C3	0.018 (2)	0.016 (2)	0.015 (2)	0.0020 (17)	−0.0041 (17)	−0.0072 (17)
C1	0.019 (2)	0.014 (2)	0.019 (2)	−0.0005 (18)	0.0015 (18)	−0.0113 (19)
C4	0.0133 (19)	0.015 (2)	0.012 (2)	−0.0005 (16)	0.0005 (15)	−0.0090 (17)

Geometric parameters (Å, º)

Br1—C1	1.887 (4)	C5—C4	1.387 (6)
O1—N1	1.220 (5)	C5—H5	0.9500
O2—N1	1.226 (5)	C2—C3	1.379 (6)

N1—C4	1.464 (5)	C2—C1	1.390 (6)
C6—C1	1.384 (6)	C2—H2	0.9500
C6—C5	1.381 (6)	C3—C4	1.380 (6)
C6—H6	0.9500	C3—H3	0.9500
O1—N1—O2	123.6 (4)	C1—C2—H2	120.6
O1—N1—C4	117.9 (4)	C4—C3—C2	119.6 (4)
O2—N1—C4	118.4 (4)	C4—C3—H3	120.2
C1—C6—C5	119.5 (4)	C2—C3—H3	120.2
C1—C6—H6	120.2	C6—C1—C2	121.5 (4)
C5—C6—H6	120.2	C6—C1—Br1	119.2 (3)
C4—C5—C6	118.7 (4)	C2—C1—Br1	119.3 (3)
C4—C5—H5	120.6	C3—C4—C5	121.8 (4)
C6—C5—H5	120.6	C3—C4—N1	119.7 (4)
C3—C2—C1	118.8 (4)	C5—C4—N1	118.4 (4)
C3—C2—H2	120.6		
C1—C6—C5—C4	0.5 (7)	C2—C3—C4—N1	−179.8 (4)
C1—C2—C3—C4	1.1 (7)	C6—C5—C4—C3	0.7 (7)
C5—C6—C1—C2	−0.9 (7)	C6—C5—C4—N1	179.0 (4)
C5—C6—C1—Br1	177.9 (3)	O1—N1—C4—C3	4.1 (6)
C3—C2—C1—C6	0.1 (7)	O2—N1—C4—C3	−175.3 (4)
C3—C2—C1—Br1	−178.7 (3)	O1—N1—C4—C5	−174.3 (4)
C2—C3—C4—C5	−1.5 (7)	O2—N1—C4—C5	6.3 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C3—H3···O1^{i}	0.95	2.52	3.359 (6)	147
C5—H5···O2^{ii}	0.95	2.54	3.276 (6)	135

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