

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

1-Bromo-2-[(E)-2-nitroethenyl]benzene

Pei-Hua Zhao,^{a*} Zhan-Heng Feng,^b Mei Zhang,^a Ya-Qing Liu^a and Gui-Zhe Zhao^a^aResearch Center for Engineering Technology of Polymeric Composites of Shanxi Province, College of Materials Science and Engineering, North University of China, Taiyuan 030051, People's Republic of China, and ^bCollege of Chemistry, Nankai University, Tianjin 300071, People's Republic of China

Correspondence e-mail: zph2004@yahoo.com.cn

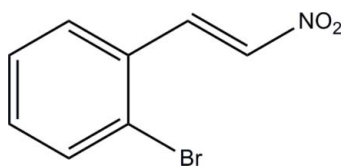
Received 26 November 2011; accepted 27 November 2011

Key indicators: single-crystal X-ray study; $T = 113$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.024; wR factor = 0.056; data-to-parameter ratio = 17.8.

In the title compound, $\text{C}_8\text{H}_6\text{BrNO}_2$, the dihedral angle between the planes of the benzene ring and the nitro group is $22.99(12)^\circ$. In the crystal, inversion dimers associated by pairs of short $\text{Br} \cdots \text{O}$ contacts [$3.2319(17)$ Å] occur.

Related literature

For background to nitro-olefins and their synthetic applications, see: Barret & Graboski (1986); Berner *et al.* (2002); Ballini *et al.* (1992).



Experimental

Crystal data

 $\text{C}_8\text{H}_6\text{BrNO}_2$ $M_r = 228.05$

Monoclinic, $P2_1/c$
 $a = 6.9570(18)$ Å
 $b = 15.646(2)$ Å
 $c = 7.9470(13)$ Å
 $\beta = 109.336(5)^\circ$
 $V = 816.2(3)$ Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 4.99$ mm⁻¹
 $T = 113$ K
 $0.20 \times 0.18 \times 0.16$ mm

Data collection

Rigaku Saturn724 CCD
 diffractometer
 Absorption correction: multi-scan
 (*CrystalClear*; Rigaku/MS, 2005)
 $T_{\min} = 0.435$, $T_{\max} = 0.502$

10346 measured reflections
 1945 independent reflections
 1466 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$
 $wR(F^2) = 0.056$
 $S = 1.08$
 1945 reflections

109 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.33$ e Å⁻³
 $\Delta\rho_{\min} = -0.78$ e Å⁻³

Data collection: *CrystalClear* (Rigaku/MS, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *CrystalStructure* (Rigaku/MS, 2005).

This work was supported financially by the Start-up Foundation of North University of China and the Youth Foundation of North University of China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6539).

References

- Ballini, R., Castagnani, R. & Petrini, M. (1992). *J. Org. Chem.* **57**, 2160–2162.
 Barret, A. G. M. & Graboski, G. G. (1986). *Chem. Rev.* **86**, 751–762.
 Berner, O. M., Tedeschi, L. & Enders, D. (2002). *Eur. J. Org. Chem.* pp. 1877–1894.
 Rigaku/MS (2005). *CrystalClear*. Rigaku/MS Inc. The Woodlands, Texas, USA.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2011). E67, o3505 [doi:10.1107/S1600536811050963]

1-Bromo-2-[(*E*)-2-nitroethenyl]benzene

P.-H. Zhao, Z.-H. Feng, M. Zhang, Y.-Q. Liu and G.-Z. Zhao

Comment

Nitro-olefins are useful building blocks in organic synthesis (Barret *et al.*, 1986). Furthermore, the character of these compounds as electron-deficient alkenes allows easy 1,4-addition reactions and this opens the way to synthetically useful C—C and C—X ($X = N, O$) bond-forming reactions (Berner *et al.* (2002), Ballini *et al.* (1992)). The title compound, (I), belongs to the class of functionalized nitroolefins.

As shown in Fig. 1, the dihedral angle between carbon double bond and phenyl groups is $12.2(2)^\circ$. As shown in Fig. 2, the crystal packing shows the weak $O\cdots Br$ intermolecular interactions.

Experimental

2-Bromobenzaldehyde (39.8 mmol, 7.36 g), nitromethane (99.2 mmol, 5.38 ml), and methanol (16.80 ml) are added to a 3-neck round bottomed flask and cooled to zero degree centigrade. While maintaining the internal reaction temperature between zero and ten degrees centigrade, aqueous 1M NaOH (100.2 mmol, 100.20 ml) is added by an addition funnel and the mixture is stirred for 15 min. Ice water mixture (70.00 ml) is added and the reaction is stirred at zero degree centigrade for 30 min. The reaction mixture is slowly added to aqueous 8M HCl (536.0 mmol, 67.00 ml) and allowed to stir until the reaction is confirmed complete by TLC. The reaction mixture is filtered and recrystallized from ethanol to give the product. Colourless prisms of (I) were obtained by slow evaporation of the dichloromethane/n-hexane solutions at room temperature.

Refinement

All the H atoms were positioned geometrically ($C-H = 0.95 \text{ \AA}$) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

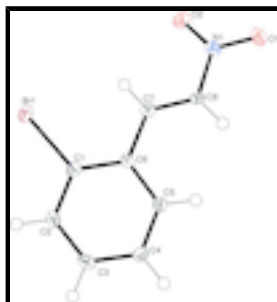


Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

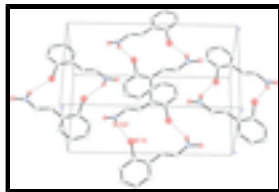


Fig. 2. The crystal packing for (I).

1-Bromo-2-[(E)-2-nitroethenyl]benzene

Crystal data

$C_8H_6BrNO_2$

$M_r = 228.05$

Monoclinic, $P2_1/c$

$a = 6.9570$ (18) Å

$b = 15.646$ (2) Å

$c = 7.9470$ (13) Å

$\beta = 109.336$ (5)°

$V = 816.2$ (3) Å³

$Z = 4$

$F(000) = 448$

$D_x = 1.856$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2970 reflections

$\theta = 2.6$ – 28.0 °

$\mu = 4.99$ mm⁻¹

$T = 113$ K

Prism, colorless

$0.20 \times 0.18 \times 0.16$ mm

Data collection

Rigaku Saturn724 CCD
diffractometer

Radiation source: rotating anode
multilayer

Detector resolution: 14.22 pixels mm⁻¹

ω and φ scans

Absorption correction: multi-scan
(*CrystalClear*; Rigaku/MS, 2005)

$T_{\min} = 0.435$, $T_{\max} = 0.502$

10346 measured reflections

1945 independent reflections

1466 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.042$

$\theta_{\max} = 27.8$ °, $\theta_{\min} = 2.6$ °

$h = -9 \rightarrow 9$

$k = -20 \rightarrow 20$

$l = -10 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.024$

$wR(F^2) = 0.056$

$S = 1.08$

1945 reflections

109 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

$w = 1/[\sigma^2(F_o^2) + (0.020P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.003$

$\Delta\rho_{\max} = 0.33$ e Å⁻³

$\Delta\rho_{\min} = -0.78$ e Å⁻³

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.39712 (4)	0.607142 (14)	0.81215 (3)	0.02051 (8)
O1	0.1113 (2)	0.21839 (10)	0.6270 (2)	0.0218 (4)
O2	0.3362 (2)	0.29119 (10)	0.8336 (2)	0.0238 (4)
N1	0.2150 (3)	0.28344 (11)	0.6807 (2)	0.0153 (4)
C1	0.3043 (3)	0.58570 (14)	0.5620 (3)	0.0141 (5)
C2	0.2914 (3)	0.65593 (14)	0.4508 (3)	0.0153 (5)
H2	0.3316	0.7109	0.5007	0.018*
C3	0.2203 (3)	0.64552 (14)	0.2684 (3)	0.0179 (5)
H3	0.2093	0.6935	0.1925	0.021*
C4	0.1644 (3)	0.56451 (15)	0.1954 (3)	0.0190 (5)
H4	0.1156	0.5571	0.0696	0.023*
C5	0.1804 (3)	0.49484 (14)	0.3071 (3)	0.0160 (5)
H5	0.1438	0.4398	0.2561	0.019*
C6	0.2490 (3)	0.50341 (13)	0.4930 (3)	0.0119 (4)
C7	0.2672 (3)	0.42903 (13)	0.6100 (3)	0.0132 (5)
H7	0.3405	0.4360	0.7334	0.016*
C8	0.1882 (3)	0.35266 (13)	0.5549 (3)	0.0135 (5)
H8	0.1138	0.3435	0.4324	0.016*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02929 (14)	0.01733 (14)	0.01372 (13)	-0.00098 (11)	0.00552 (10)	-0.00406 (9)
O1	0.0270 (10)	0.0133 (8)	0.0233 (9)	-0.0033 (7)	0.0058 (8)	0.0004 (7)
O2	0.0272 (10)	0.0228 (9)	0.0148 (9)	0.0010 (7)	-0.0020 (7)	0.0036 (7)
N1	0.0178 (10)	0.0113 (10)	0.0182 (10)	0.0032 (8)	0.0079 (8)	0.0012 (8)
C1	0.0107 (11)	0.0190 (12)	0.0120 (11)	0.0007 (9)	0.0032 (9)	-0.0014 (9)
C2	0.0151 (12)	0.0119 (12)	0.0207 (13)	-0.0022 (9)	0.0081 (10)	-0.0021 (9)
C3	0.0178 (12)	0.0158 (12)	0.0207 (13)	-0.0014 (10)	0.0074 (10)	0.0057 (10)
C4	0.0211 (13)	0.0227 (14)	0.0122 (12)	-0.0046 (10)	0.0042 (10)	-0.0008 (10)
C5	0.0173 (12)	0.0146 (12)	0.0161 (12)	-0.0010 (9)	0.0056 (10)	-0.0023 (9)
C6	0.0099 (11)	0.0113 (11)	0.0148 (12)	0.0014 (8)	0.0046 (9)	0.0015 (9)

supplementary materials

C7	0.0119 (11)	0.0150 (12)	0.0124 (11)	0.0032 (9)	0.0037 (9)	0.0005 (9)
C8	0.0164 (12)	0.0132 (12)	0.0111 (12)	0.0047 (9)	0.0050 (9)	0.0037 (9)

Geometric parameters (Å, °)

Br1—C1	1.906 (2)	C3—H3	0.9500
O1—N1	1.239 (2)	C4—C5	1.387 (3)
O2—N1	1.234 (2)	C4—H4	0.9500
N1—C8	1.444 (3)	C5—C6	1.401 (3)
C1—C2	1.394 (3)	C5—H5	0.9500
C1—C6	1.402 (3)	C6—C7	1.468 (3)
C2—C3	1.377 (3)	C7—C8	1.328 (3)
C2—H2	0.9500	C7—H7	0.9500
C3—C4	1.395 (3)	C8—H8	0.9500
O2—N1—O1	123.39 (19)	C3—C4—H4	120.1
O2—N1—C8	119.86 (18)	C4—C5—C6	121.8 (2)
O1—N1—C8	116.75 (18)	C4—C5—H5	119.1
C2—C1—C6	121.6 (2)	C6—C5—H5	119.1
C2—C1—Br1	116.81 (16)	C1—C6—C5	116.97 (19)
C6—C1—Br1	121.57 (16)	C1—C6—C7	121.70 (19)
C3—C2—C1	119.9 (2)	C5—C6—C7	121.31 (19)
C3—C2—H2	120.0	C8—C7—C6	124.4 (2)
C1—C2—H2	120.0	C8—C7—H7	117.8
C2—C3—C4	119.9 (2)	C6—C7—H7	117.8
C2—C3—H3	120.0	C7—C8—N1	120.14 (19)
C4—C3—H3	120.0	C7—C8—H8	119.9
C5—C4—C3	119.7 (2)	N1—C8—H8	119.9
C5—C4—H4	120.1		
C6—C1—C2—C3	0.9 (3)	Br1—C1—C6—C7	-2.4 (3)
Br1—C1—C2—C3	-178.25 (16)	C4—C5—C6—C1	-1.0 (3)
C1—C2—C3—C4	-1.0 (3)	C4—C5—C6—C7	-179.4 (2)
C2—C3—C4—C5	0.2 (3)	C1—C6—C7—C8	168.8 (2)
C3—C4—C5—C6	0.9 (4)	C5—C6—C7—C8	-13.0 (3)
C2—C1—C6—C5	0.1 (3)	C6—C7—C8—N1	179.9 (2)
Br1—C1—C6—C5	179.22 (16)	O2—N1—C8—C7	-10.9 (3)
C2—C1—C6—C7	178.5 (2)	O1—N1—C8—C7	168.8 (2)

Fig. 1

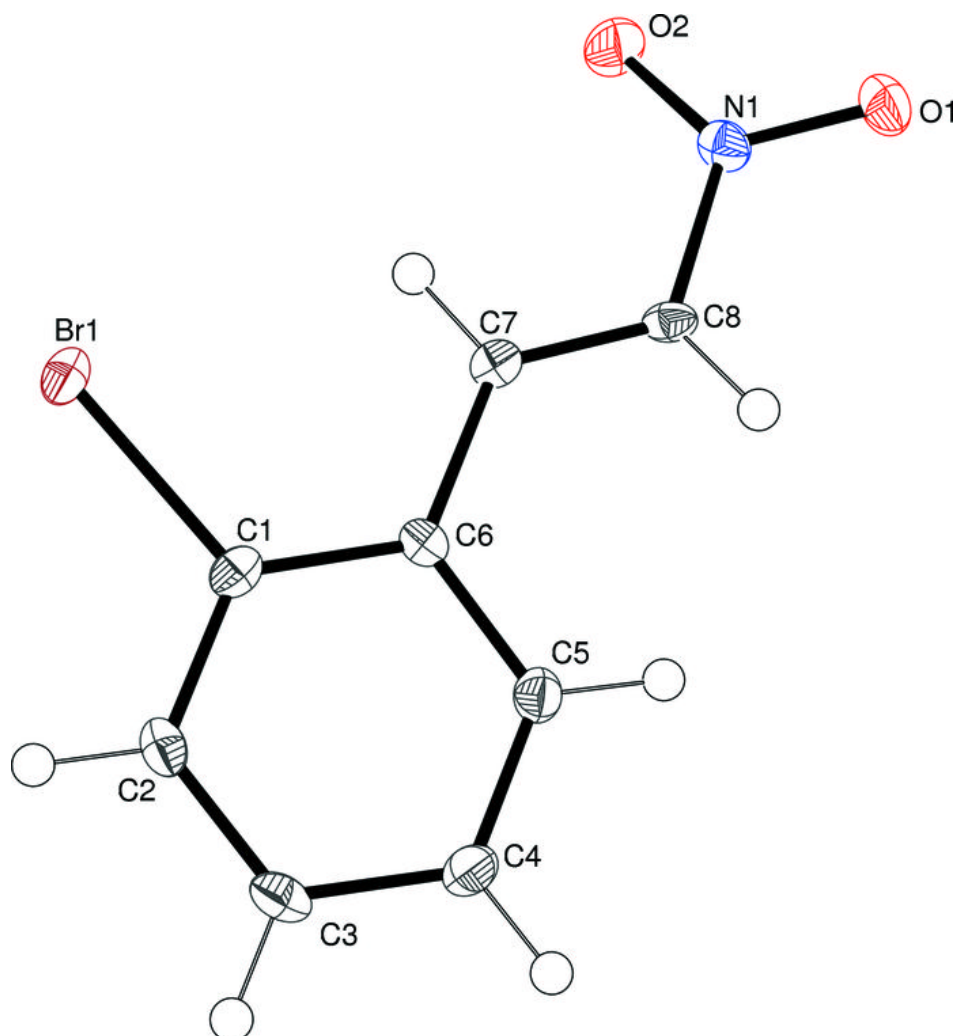


Fig. 2

