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(E)-1-Bromo-4-(2-nitroprop-1-enyl)-benzene

Bailin Li

Department of Pharmaceutical and Chemical Engineering, Taizhou College, Linhai, Zhejiang 317000, People's Republic of China
Correspondence e-mail: bailinli1972@gmail.com

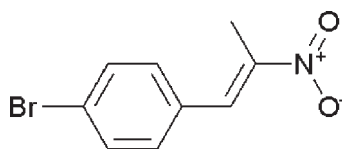
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.035; wR factor = 0.094; data-to-parameter ratio = 17.6.

The title compound, $\text{C}_9\text{H}_8\text{BrNO}_2$, which was synthesized by the condensation of 4-bromobenzaldehyde with nitroethane, possesses a *trans* configuration. The dihedral angle between the benzene ring and the mean plane of the double bond is 7.31 (3)°. The crystal structure is stabilized by short intermolecular $\text{Br} \cdots \text{O}$ contacts [3.168 (4) Å].

Related literature

For general background to nitroalkenes as intermediates in the preparation of numerous products including insecticides and pharmacologically active substances, see: Boelle *et al.* (1998); Vallejos *et al.* (2005). For related structures, see: Boys *et al.* (1993); Mugnoli *et al.* (1991).



Experimental

Crystal data

$\text{C}_9\text{H}_8\text{BrNO}_2$

$M_r = 242.07$

Triclinic, $P\bar{1}$
 $a = 6.9787$ (5) Å
 $b = 7.4123$ (5) Å
 $c = 9.7659$ (6) Å
 $\alpha = 105.435$ (2)°
 $\beta = 95.087$ (2)°
 $\gamma = 104.323$ (2)°

$V = 465.31$ (5) Å³
 $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 4.38$ mm⁻¹
 $T = 296$ K
 $0.21 \times 0.19 \times 0.08$ mm

Data collection

Rigaku R-Axis RAPID
diffractometer
Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)
 $T_{\min} = 0.388$, $T_{\max} = 0.703$

4605 measured reflections
2112 independent reflections
1303 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.094$
 $S = 1.00$
2112 reflections

120 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.46$ e Å⁻³
 $\Delta\rho_{\min} = -0.71$ e Å⁻³

Data collection: *PROCESS-AUTO* (Rigaku, 2006); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSK, 2007); 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); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2017).

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supplementary materials

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(*E*)-1-Bromo-4-(2-nitroprop-1-enyl)benzene

B. Li

Comment

Nitroalkenes are valuable intermediates for preparation of numerous products including insecticides and pharmacologically active substances (Boelle *et al.*, 1998 and Vallejos *et al.*, 2005) in which the nitro group can be easily transformed into a variety of groups with different functionalities, such as amine, carbonyl groups, *etc.*. In this article, the crystal structure of the title compound (*E*)-1-bromo-4-(2-nitroprop-1-enyl)benzene is presented (Fig. 1). The dihedral angle between the benzene ring and the mean plan of the double bond H7/C7/C8/C9 is 7.31 (3)°. The crystal structure is stabilized by short intermolecular Br—O contacts [3.168 (4) Å].

Experimental

To a solution of 4-bromobenzaldehyde (50 mmol) in AcOH (25 ml), nitroethane (75 mmol) was added, followed by butylamine (100 mmol, 7.4 ml). The mixture was sonicated at 333 K, until TLC showed full conversion of aldehyde. The mixture was poured into ice water, the precipitate was filtered off, washed with water and recrystallized from EtOH to give (*E*)-1-bromo-4-(2-nitroprop-1-enyl)benzene. Suitable crystals of the title compound were obtained by slow evaporation of an ethanol solution at room temperature.

Refinement

All carbon-bonded H atoms were placed in calculated positions with C—H = 0.93 Å (aromatic), C—H = 0.96 Å (*sp*) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2_{\text{eq}}(\text{C})$.

Figures

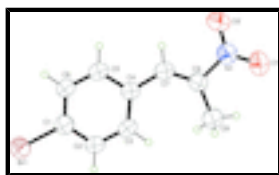


Fig. 1. The asymmetric unit of the title compound (I) with the atomic labeling scheme. Displacement ellipsoids are drawn at the 40% probability level.

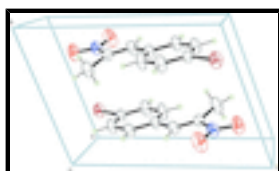


Fig. 2. Molecular packing of the title compound (I) viewed down the *a* axis.

(E)-1-Bromo-4-(2-nitroprop-1-enyl)benzene

Crystal data

$C_9H_8BrNO_2$	$Z = 2$
$M_r = 242.07$	$F_{000} = 240$
Triclinic, $P\bar{1}$	$D_x = 1.728 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 6.9787 (5) \text{ \AA}$	Cell parameters from 3184 reflections
$b = 7.4123 (5) \text{ \AA}$	$\theta = 3.1\text{--}27.4^\circ$
$c = 9.7659 (6) \text{ \AA}$	$\mu = 4.38 \text{ mm}^{-1}$
$\alpha = 105.435 (2)^\circ$	$T = 296 \text{ K}$
$\beta = 95.087 (2)^\circ$	Platelet, yellow
$\gamma = 104.323 (2)^\circ$	$0.21 \times 0.19 \times 0.08 \text{ mm}$
$V = 465.31 (5) \text{ \AA}^3$	

Data collection

Rigaku R-Axis RAPID diffractometer	2112 independent reflections
Radiation source: rolling anode	1303 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.027$
Detector resolution: $10.00 \text{ pixels mm}^{-1}$	$\theta_{\text{max}} = 27.4^\circ$
$T = 296 \text{ K}$	$\theta_{\text{min}} = 3.1^\circ$
ω scans	$h = -9 \rightarrow 9$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$k = -9 \rightarrow 8$
$T_{\text{min}} = 0.388$, $T_{\text{max}} = 0.703$	$l = -12 \rightarrow 12$
4605 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.035$	$w = 1/[\sigma^2(F_o^2) + (0.012P)^2 + 0.950P]$
$wR(F^2) = 0.094$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{\text{max}} < 0.001$
2112 reflections	$\Delta\rho_{\text{max}} = 0.46 \text{ e \AA}^{-3}$
120 parameters	$\Delta\rho_{\text{min}} = -0.71 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.0149 (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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.90197 (7)	0.24927 (9)	0.09032 (5)	0.0754 (2)
N1	0.2076 (6)	0.2311 (6)	0.7592 (4)	0.0660 (10)
O1	0.2341 (5)	0.2609 (6)	0.8891 (4)	0.0937 (12)
O2	0.0411 (5)	0.1719 (7)	0.6875 (4)	0.1038 (14)
C8	0.3870 (6)	0.2659 (6)	0.6875 (4)	0.0514 (9)
C1	0.7334 (6)	0.2408 (6)	0.2327 (4)	0.0567 (10)
C5	0.4097 (6)	0.1824 (7)	0.2989 (4)	0.0631 (12)
H5	0.2712	0.1456	0.2720	0.076*
C7	0.3513 (6)	0.2154 (7)	0.5461 (4)	0.0594 (11)
H7	0.2165	0.1633	0.5051	0.071*
C4	0.4907 (6)	0.2283 (6)	0.4430 (4)	0.0524 (10)
C3	0.6976 (6)	0.2770 (8)	0.4767 (5)	0.0776 (15)
H3	0.7569	0.3055	0.5720	0.093*
C9	0.5777 (7)	0.3501 (9)	0.7900 (5)	0.0825 (16)
H9A	0.6349	0.2476	0.7987	0.099*
H9B	0.5528	0.4165	0.8825	0.099*
H9C	0.6694	0.4409	0.7555	0.099*
C2	0.8179 (6)	0.2840 (8)	0.3723 (5)	0.0742 (14)
H2	0.9565	0.3183	0.3976	0.089*
C6	0.5299 (6)	0.1901 (7)	0.1943 (4)	0.0680 (13)
H6	0.4727	0.1609	0.0985	0.082*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0560 (3)	0.1058 (5)	0.0598 (3)	0.0136 (2)	0.0153 (2)	0.0237 (3)
N1	0.055 (2)	0.081 (3)	0.055 (2)	0.0147 (19)	0.0103 (18)	0.014 (2)
O1	0.076 (2)	0.142 (4)	0.056 (2)	0.019 (2)	0.0205 (17)	0.025 (2)
O2	0.0467 (19)	0.173 (4)	0.074 (2)	0.017 (2)	0.0114 (17)	0.020 (2)
C8	0.045 (2)	0.056 (3)	0.050 (2)	0.0112 (18)	0.0092 (17)	0.0142 (19)
C1	0.052 (2)	0.066 (3)	0.052 (2)	0.013 (2)	0.0100 (19)	0.019 (2)
C5	0.042 (2)	0.087 (3)	0.050 (2)	0.012 (2)	-0.0015 (18)	0.012 (2)

supplementary materials

C7	0.043 (2)	0.077 (3)	0.051 (2)	0.015 (2)	0.0032 (17)	0.012 (2)
C4	0.042 (2)	0.064 (3)	0.048 (2)	0.0140 (19)	0.0039 (16)	0.013 (2)
C3	0.050 (2)	0.134 (5)	0.044 (2)	0.024 (3)	-0.0013 (19)	0.022 (3)
C9	0.052 (3)	0.122 (5)	0.055 (3)	0.008 (3)	0.003 (2)	0.015 (3)
C2	0.038 (2)	0.123 (4)	0.055 (3)	0.018 (2)	0.0015 (19)	0.022 (3)
C6	0.047 (2)	0.103 (4)	0.045 (2)	0.013 (2)	0.0013 (18)	0.017 (2)

Geometric parameters (Å, °)

Br1—C1	1.902 (4)	C7—C4	1.466 (5)
N1—O2	1.214 (5)	C7—H7	0.9300
N1—O1	1.217 (4)	C4—C3	1.385 (6)
N1—C8	1.488 (5)	C3—C2	1.380 (6)
C8—C7	1.314 (5)	C3—H3	0.9300
C8—C9	1.478 (6)	C9—H9A	0.9600
C1—C2	1.357 (6)	C9—H9B	0.9600
C1—C6	1.366 (6)	C9—H9C	0.9600
C5—C6	1.381 (6)	C2—H2	0.9300
C5—C4	1.388 (5)	C6—H6	0.9300
C5—H5	0.9300		
O2—N1—O1	122.1 (4)	C5—C4—C7	117.7 (4)
O2—N1—C8	119.7 (4)	C2—C3—C4	121.6 (4)
O1—N1—C8	118.2 (4)	C2—C3—H3	119.2
C7—C8—C9	130.9 (4)	C4—C3—H3	119.2
C7—C8—N1	115.8 (4)	C8—C9—H9A	109.5
C9—C8—N1	113.2 (3)	C8—C9—H9B	109.5
C2—C1—C6	120.5 (4)	H9A—C9—H9B	109.5
C2—C1—Br1	119.2 (3)	C8—C9—H9C	109.5
C6—C1—Br1	120.3 (3)	H9A—C9—H9C	109.5
C6—C5—C4	121.6 (4)	H9B—C9—H9C	109.5
C6—C5—H5	119.2	C1—C2—C3	119.9 (4)
C4—C5—H5	119.2	C1—C2—H2	120.0
C8—C7—C4	130.1 (4)	C3—C2—H2	120.0
C8—C7—H7	115.0	C1—C6—C5	119.5 (4)
C4—C7—H7	115.0	C1—C6—H6	120.2
C3—C4—C5	116.9 (4)	C5—C6—H6	120.2
C3—C4—C7	125.4 (4)		
O2—N1—C8—C7	-4.8 (6)	C8—C7—C4—C5	-173.5 (5)
O1—N1—C8—C7	174.2 (5)	C5—C4—C3—C2	1.5 (8)
O2—N1—C8—C9	176.1 (5)	C7—C4—C3—C2	179.4 (5)
O1—N1—C8—C9	-4.9 (6)	C6—C1—C2—C3	-0.1 (8)
C9—C8—C7—C4	-0.7 (9)	Br1—C1—C2—C3	-179.5 (4)
N1—C8—C7—C4	-179.7 (4)	C4—C3—C2—C1	-0.6 (8)
C6—C5—C4—C3	-1.8 (7)	C2—C1—C6—C5	-0.2 (8)
C6—C5—C4—C7	-179.8 (4)	Br1—C1—C6—C5	179.2 (4)
C8—C7—C4—C3	8.7 (8)	C4—C5—C6—C1	1.1 (8)

Fig. 1

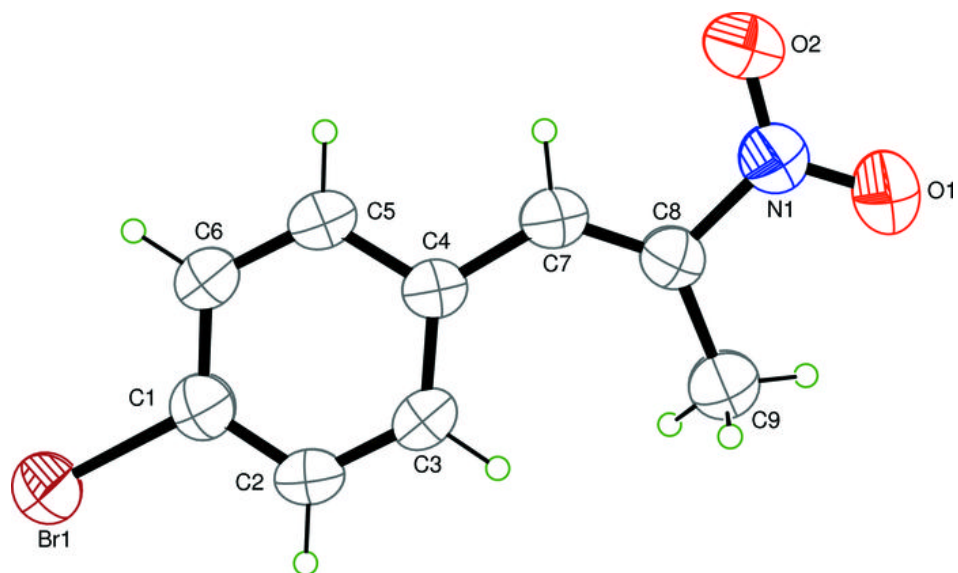


Fig. 2

