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(*E*)-1-Nitro-2-(2-nitroprop-1-enyl)-benzene

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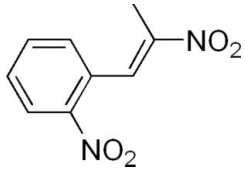
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.004 Å; R factor = 0.059; wR factor = 0.135; data-to-parameter ratio = 12.6.

The title compound, $C_9H_8N_2O_4$, adopts an *E* conformation about the C=C bond. The $CH_{phenyl}-C_{phenyl}-CH-C(-NO_2)$ torsion angle is −57.7 (3)°. The crystal structure features weak intermolecular C—H···O interactions.

Related literature

For background to nitroalkenes, see: Ballini & Petrini (2004); Berner *et al.* (2002); Ono (2001).



Experimental

Crystal data

$C_9H_8N_2O_4$
M_r = 208.17
Monoclinic, $P2_1/n$
a = 6.8274 (9) Å
b = 15.5666 (12) Å
c = 9.9045 (10) Å
β = 113.202 (3)°
V = 967.51 (18) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 296 K
0.58 × 0.46 × 0.32 mm

Data collection

Rigaku R-AXIS RAPID/ZJUG diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.932, T_{max} = 0.964
7450 measured reflections
1736 independent reflections
1193 reflections with $I > 2\sigma(I)$
R_{int} = 0.072

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.135
S = 1.00
1736 reflections
138 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O3^{i}	0.93	2.60	3.163 (5)	119
C9—H9A···O2^{ii}	0.96	2.70	3.403 (4)	131

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

Data collection: *PROCESS-AUTO* (Rigaku, 2006); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku, 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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5280).

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(*E*)-1-Nitro-2-(2-nitroprop-1-enyl)benzene

Li-Li Shen, Zhao-Bo Li and Jia-Jia Li

S1. Comment

Nitroalkenes are important organic intermediates, since they can be converted to synthetically useful N– and O-containing organic molecules, such as amines, aldehydes, carboxylic acids, or denitrated compounds (Ono, 2001; Berner *et al.*, 2002; Ballini & Petrini, 2004). As a contribution in this field, we have synthesized a series of nitroalkenes by employing benzaldehydes and nitroethane. We report here one of this nitroalkenes, *i.e.* the title compound. The C7═C8 bond involves the *E* configuration with the C3—C2—C7—C8 torsion angle of -57.7 (3)° (Fig. 1). The conformation of (I) is stabilized by weak intermolecular C6—H6···O3'and C9—H9A···O2′ interactions (Fig. 2 and Table 1).

S2. Experimental

To a solution of 2-nitrobenzaldehyde (50 mmol) in AcOH (25 mL), nitroethane (75 mmol) was added, followed by butylamine (100 mmol, 7.4 mL). The mixture was sonicated at 60 °C, until GC showed full conversion of the aldehyde. The mixture was poured into ice water, the precipitate was filtered off, washed with water and recrystallized from EtOH/EtOAc to give the product. Single crystals were obtained by slow evaporation of a EtOH solution of the compound.

S3. Refinement

All H atoms were placed in calculated positions and refined using a riding model, with C—H = 0.93–0.96 Å, and with U_{iso}(H) = 1.2 U_{eq}(C) or 1.5 U_{eq}(C) for methyl H atoms.

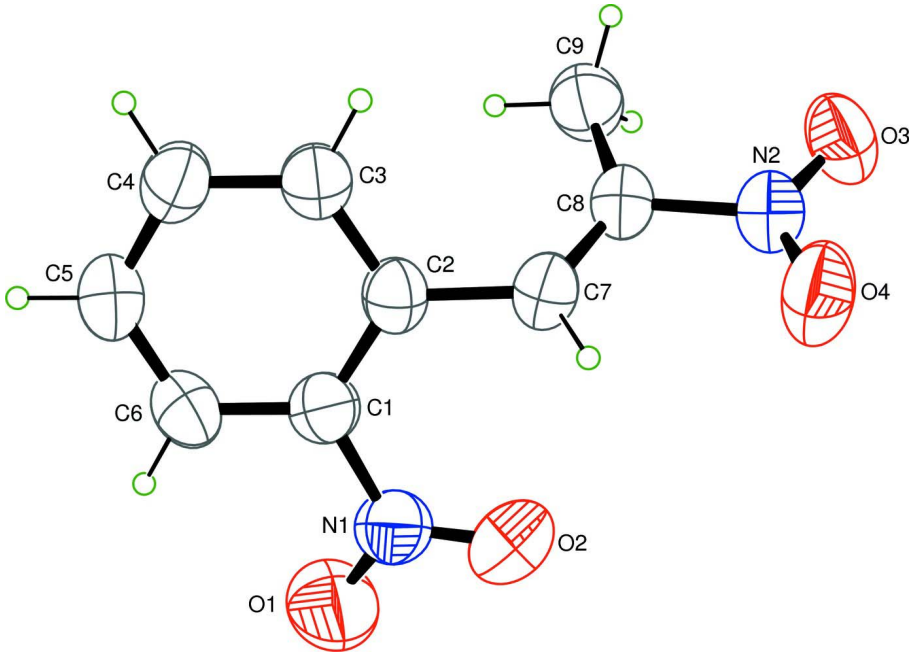

Figure 1

The asymmetric unit of the title compound with the atomic labeling scheme; displacement ellipsoids are drawn at the 50% probability level.

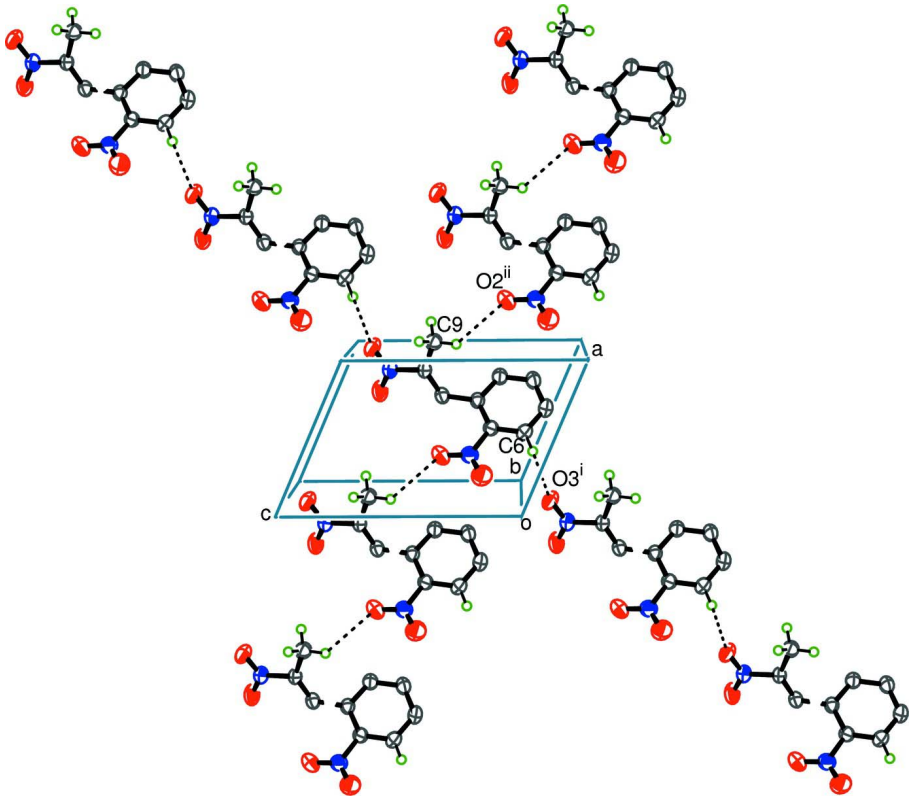

Figure 2

The view of intermolecular interactions illustrated as dash lines.

(*E*)-1-Nitro-2-(2-nitroprop-1-enyl)benzene

Crystal data

$C_9H_8N_2O_4$
$M_r = 208.17$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 6.8274$ (9) Å
$b = 15.5666$ (12) Å
$c = 9.9045$ (10) Å
$\beta = 113.202$ (3)°
$V = 967.51$ (18) Å^3
$Z = 4$
$F(000) = 432$
$D_x = 1.429$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 5183 reflections
$\theta = 3.2$–$27.5°$
$\mu = 0.12$ mm^{-1}
$T = 296$ K
Chunk, yellow
0.58 × 0.46 × 0.32 mm

Data collection

Rigaku R-AXIS RAPID/ZJUG diffractometer
Radiation source: rotating anode
Graphite monochromator
Detector resolution: 10.00 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.932$, $T_{max} = 0.964$
7450 measured reflections
1736 independent reflections
1193 reflections with $I > 2\sigma(I)$
$R_{int} = 0.072$
$\theta_{max} = 25.2°$, $\theta_{min} = 3.4°$
$h = -7 \rightarrow 8$
$k = -18 \rightarrow 18$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.135$
$S = 1.00$
1736 reflections
138 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
$w = 1/[\sigma^2(F_o^2) + (0.019P)^2 + 0.5218P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.170 (12)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.5466 (4)	0.10207 (16)	0.2728 (3)	0.0619 (6)
C2	0.7192 (4)	0.14601 (15)	0.3747 (3)	0.0601 (6)
C3	0.8689 (4)	0.17747 (17)	0.3231 (3)	0.0709 (7)
H3	0.9876	0.2069	0.3873	0.085*
C4	0.8450 (5)	0.16585 (18)	0.1791 (3)	0.0774 (8)
H4	0.9474	0.1873	0.1480	0.093*
C5	0.6713 (5)	0.12290 (18)	0.0817 (3)	0.0782 (8)
H5	0.6548	0.1159	−0.0154	0.094*
C6	0.5215 (4)	0.09026 (17)	0.1286 (3)	0.0713 (7)
H6	0.4042	0.0604	0.0637	0.086*
C7	0.7489 (4)	0.16389 (15)	0.5287 (3)	0.0633 (7)
H7	0.6399	0.1925	0.5440	0.076*
C8	0.9185 (4)	0.14215 (15)	0.6453 (3)	0.0597 (6)
C9	1.1075 (4)	0.09071 (19)	0.6571 (3)	0.0789 (8)
H9A	1.0943	0.0736	0.5608	0.118*
H9B	1.1164	0.0405	0.7156	0.118*
H9C	1.2339	0.1247	0.7026	0.118*
N1	0.3819 (4)	0.06343 (17)	0.3147 (3)	0.0799 (7)
N2	0.9179 (4)	0.16809 (14)	0.7890 (2)	0.0702 (6)
O1	0.2593 (4)	0.01245 (18)	0.2313 (3)	0.1285 (10)
O2	0.3754 (4)	0.08268 (17)	0.4315 (3)	0.1097 (8)
O3	1.0659 (3)	0.14555 (14)	0.8995 (2)	0.0925 (7)
O4	0.7722 (4)	0.21184 (15)	0.7936 (2)	0.1023 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0613 (14)	0.0631 (15)	0.0609 (15)	0.0036 (12)	0.0238 (12)	0.0103 (11)
C2	0.0654 (14)	0.0611 (14)	0.0548 (13)	0.0078 (12)	0.0248 (12)	0.0053 (11)
C3	0.0736 (16)	0.0778 (17)	0.0626 (16)	−0.0069 (14)	0.0282 (13)	−0.0023 (13)
C4	0.0854 (18)	0.089 (2)	0.0661 (16)	−0.0078 (15)	0.0387 (15)	0.0021 (14)
C5	0.093 (2)	0.0868 (19)	0.0555 (15)	0.0000 (16)	0.0296 (15)	0.0035 (13)
C6	0.0718 (16)	0.0734 (17)	0.0591 (16)	0.0003 (13)	0.0155 (13)	0.0031 (13)
C7	0.0715 (15)	0.0649 (15)	0.0604 (15)	0.0069 (12)	0.0335 (13)	0.0019 (12)
C8	0.0699 (15)	0.0588 (14)	0.0530 (14)	−0.0005 (12)	0.0271 (12)	−0.0022 (11)
C9	0.0728 (17)	0.089 (2)	0.0725 (17)	0.0113 (14)	0.0258 (14)	−0.0033 (14)
N1	0.0720 (15)	0.0871 (17)	0.0793 (17)	−0.0004 (13)	0.0284 (13)	0.0154 (13)
N2	0.0839 (15)	0.0681 (14)	0.0606 (13)	0.0013 (12)	0.0305 (12)	−0.0016 (10)
O1	0.1249 (18)	0.146 (2)	0.1092 (19)	−0.0697 (18)	0.0400 (15)	−0.0081 (16)
O2	0.1001 (16)	0.144 (2)	0.1069 (18)	−0.0120 (14)	0.0642 (14)	−0.0012 (15)
O3	0.0962 (14)	0.1094 (16)	0.0554 (11)	0.0055 (12)	0.0120 (10)	−0.0029 (10)
O4	0.1208 (17)	0.1218 (18)	0.0747 (13)	0.0400 (15)	0.0497 (13)	0.0037 (12)

Geometric parameters (Å, º)

C1—C6	1.382 (3)	C7—C8	1.318 (3)
C1—C2	1.392 (3)	C7—H7	0.9300
C1—N1	1.472 (3)	C8—C9	1.483 (3)
C2—C3	1.400 (3)	C8—N2	1.481 (3)
C2—C7	1.483 (3)	C9—H9A	0.9600
C3—C4	1.381 (3)	C9—H9B	0.9600
C3—H3	0.9300	C9—H9C	0.9600
C4—C5	1.372 (4)	N1—O1	1.212 (3)
C4—H4	0.9300	N1—O2	1.212 (3)
C5—C6	1.376 (4)	N2—O3	1.212 (3)
C5—H5	0.9300	N2—O4	1.221 (3)
C6—H6	0.9300		
C6—C1—C2	122.6 (2)	C8—C7—C2	124.8 (2)
C6—C1—N1	116.1 (2)	C8—C7—H7	117.6
C2—C1—N1	121.3 (2)	C2—C7—H7	117.6
C3—C2—C1	116.0 (2)	C7—C8—C9	130.1 (2)
C3—C2—C7	119.1 (2)	C7—C8—N2	116.0 (2)
C1—C2—C7	124.8 (2)	C9—C8—N2	113.8 (2)
C4—C3—C2	121.7 (2)	C8—C9—H9A	109.5
C4—C3—H3	119.2	C8—C9—H9B	109.5
C2—C3—H3	119.2	H9A—C9—H9B	109.5
C5—C4—C3	120.5 (3)	C8—C9—H9C	109.5
C5—C4—H4	119.8	H9A—C9—H9C	109.5
C3—C4—H4	119.8	H9B—C9—H9C	109.5
C6—C5—C4	119.6 (2)	O1—N1—O2	122.5 (3)
C6—C5—H5	120.2	O1—N1—C1	118.3 (3)
C4—C5—H5	120.2	O2—N1—C1	119.1 (3)
C5—C6—C1	119.6 (2)	O3—N2—O4	122.0 (2)
C5—C6—H6	120.2	O3—N2—C8	118.1 (2)
C1—C6—H6	120.2	O4—N2—C8	119.9 (2)
C6—C1—C2—C3	−0.5 (4)	C1—C2—C7—C8	124.7 (3)
N1—C1—C2—C3	178.0 (2)	C2—C7—C8—C9	−5.5 (4)
C6—C1—C2—C7	177.1 (2)	C2—C7—C8—N2	178.6 (2)
N1—C1—C2—C7	−4.4 (4)	C6—C1—N1—O1	12.6 (4)
C1—C2—C3—C4	0.4 (4)	C2—C1—N1—O1	−166.0 (3)
C7—C2—C3—C4	−177.3 (2)	C6—C1—N1—O2	−168.4 (2)
C2—C3—C4—C5	0.2 (4)	C2—C1—N1—O2	13.1 (4)
C3—C4—C5—C6	−0.9 (4)	C7—C8—N2—O3	176.3 (2)
C4—C5—C6—C1	0.8 (4)	C9—C8—N2—O3	−0.3 (3)
C2—C1—C6—C5	−0.1 (4)	C7—C8—N2—O4	−4.6 (3)
N1—C1—C6—C5	−178.6 (2)	C9—C8—N2—O4	178.8 (2)
C3—C2—C7—C8	−57.7 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···O3^{i}	0.93	2.60	3.163 (5)	119
C9—H9*A*···O2^{ii}	0.96	2.70	3.403 (4)	131

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