Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

1-Methyl-4-[(1*E*)-2-nitroprop-1-en-1-yl]-benzene

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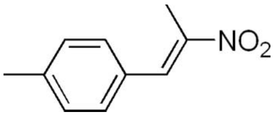
Received 8 May 2012; accepted 7 June 2012

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.045; wR factor = 0.134; data-to-parameter ratio = 17.9.

The title compound, $C_{10}H_{11}NO_2$, adopts an E conformation about the C=C bond. The C=C—C=C torsion angle is 32.5 (3). The crystal structure features weak intermolecular C—H···O interactions.

Related literature

For background to the chemistry of nitroalkenes, see: Ballini & Petrini (2004); Berner *et al.* (2002); Ono (2001). For a related structure, see: Yang *et al.* (2010).



Experimental

Crystal data

$C_{10}H_{11}NO_2$
M_r = 177.20
Orthorhombic, $Pbca$
a = 11.0610 (5) Å
b = 7.5840 (4) Å
c = 22.6420 (11) Å
V = 1899.36 (16) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 296 K
0.58 × 0.43 × 0.36 mm

Data collection

Rigaku R-AXIS-RAPID/ZJUG diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995) T_{min} = 0.941, T_{max} = 0.969
16972 measured reflections
2162 independent reflections
1325 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.134
S = 1.00
2162 reflections
121 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8···O1^{i}	0.93	2.55	3.369 (2)	147
C2—H2···O2^{ii}	0.93	2.66	3.551 (3)	162

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

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

The authors thank Mr Jianming Gu for the single-crystal X-ray analysis. They are also grateful for financial support from the State Key Laboratory Breeding Base of Green Chemistry–Synthesis Technology of Zhejiang University of Technology (GCTKF2012010).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2167).

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supporting information

Acta Cryst. (2012). E**68**, o2065   [https://doi.org/10.1107/S1600536812025913]

1-Methyl-4-[(1*E*)-2-nitroprop-1-en-1-yl]benzene

Zhao-Bo Li, Li-Li Shen and Jian-An Zheng

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 (Yang *et al.*, 2010). We report here the crystal structure of the title compound (Fig. 1).

The molecule adopts an *E* configuration with respect to the C8=C9 double bond. The torsion angle C9—C8—C1—C6 is 32.5 (3)°. In the crystal structure, the molecules interact through weak intermolecular C8—H8···O1^{i} and C2—H2···O2^{ii} hydrogen bonds (symmetry codes: *i* = -*x*+1, -*y*, -*z*+1; *ii* = *x*+1/2, -*y*+1/2, -*z*+1; Fig. 2 and Table 1).

S2. Experimental

To a solution of *p*-tolualdehyde (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 final product. Single crystals were obtained by slow evaporation of a *n*-hexane/EtOAc (10:1, *v*/*v*) solution.

S3. Refinement

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

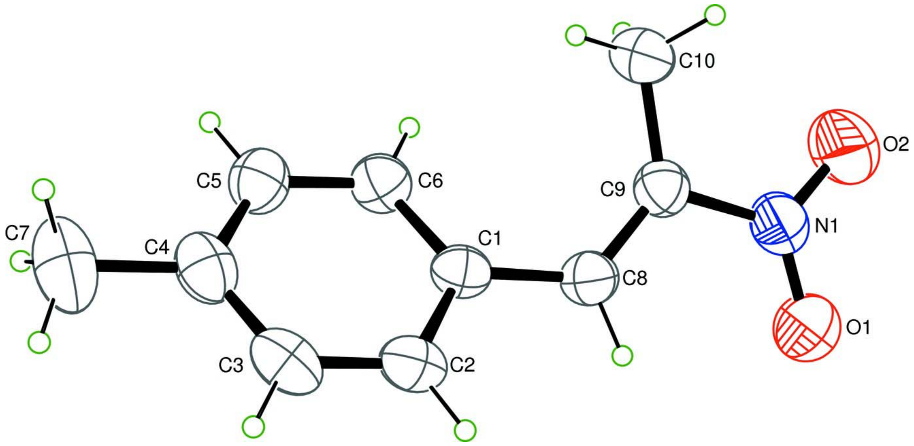

Figure 1

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

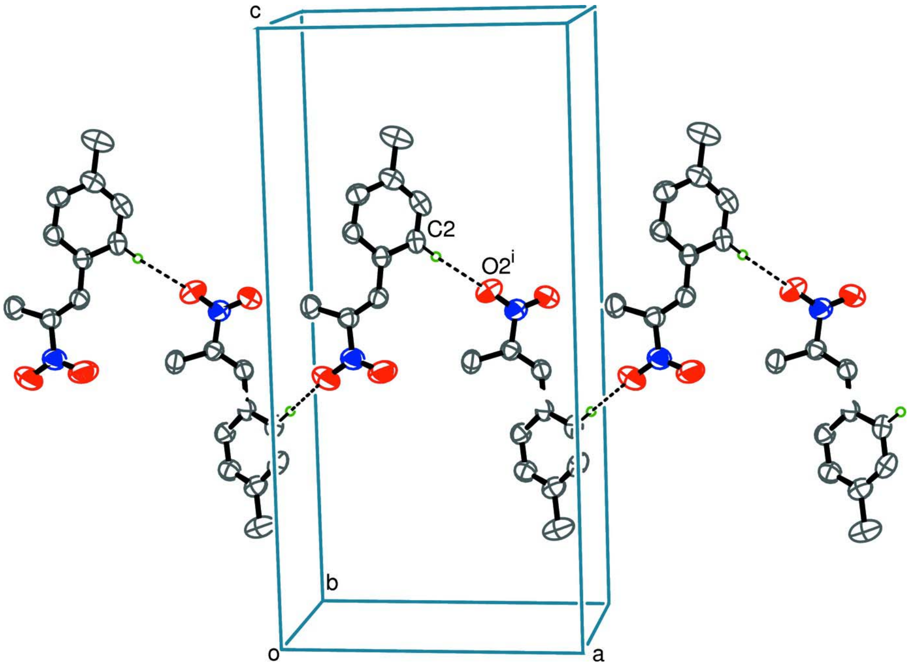

Figure 2

View of the intermolecular interactions illustrated as dashed lines.

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Crystal data

$C_{10}H_{11}NO_2$
$M_r = 177.20$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 11.0610$ (5) Å
$b = 7.5840$ (4) Å
$c = 22.6420$ (11) Å
$V = 1899.36$ (16) Å^3
$Z = 8$
$F(000) = 752$
$D_x = 1.239$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 9467 reflections
$\theta = 3.2$–27.4°
$\mu = 0.09$ mm^{-1}
$T = 296$ K
Chunk, yellow
0.58 × 0.43 × 0.36 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)
$T_{\min} = 0.941$, $T_{\max} = 0.969$
16972 measured reflections
2162 independent reflections
1325 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.033$
$\theta_{\max} = 27.4°$, $\theta_{\min} = 3.4°$
$h = -14 \rightarrow 14$
$k = -9 \rightarrow 9$
$l = -29 \rightarrow 29$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.134$
$S = 1.00$
2162 reflections
121 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

$w = 1/[\sigma^2(F_o^2) + (0.0552P)^2 + 0.4885P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0051 (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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.35993 (13)	0.1487 (2)	0.63005 (7)	0.0487 (4)
C2	0.46387 (14)	0.2326 (2)	0.65095 (8)	0.0577 (4)
H2	0.5248	0.2634	0.6246	0.069*
C3	0.47750 (16)	0.2703 (2)	0.71015 (8)	0.0643 (5)
H3	0.5464	0.3300	0.7227	0.077*
C4	0.39139 (17)	0.2218 (2)	0.75151 (8)	0.0614 (5)
C5	0.29031 (16)	0.1324 (2)	0.73072 (8)	0.0615 (5)
H5	0.2318	0.0957	0.7575	0.074*
C6	0.27416 (14)	0.0963 (2)	0.67157 (7)	0.0547 (4)
H6	0.2053	0.0363	0.6592	0.066*
C7	0.4073 (2)	0.2610 (3)	0.81617 (9)	0.0938 (7)
H7A	0.4047	0.1529	0.8382	0.141*
H7B	0.3435	0.3374	0.8292	0.141*
H7C	0.4839	0.3177	0.8223	0.141*
C8	0.34896 (14)	0.1144 (2)	0.56645 (7)	0.0531 (4)
H8	0.4211	0.0963	0.5462	0.064*
C9	0.24878 (15)	0.1058 (2)	0.53415 (7)	0.0524 (4)
C10	0.12094 (14)	0.1404 (3)	0.55103 (8)	0.0676 (5)
H10A	0.0791	0.0304	0.5557	0.101*
H10B	0.0824	0.2087	0.5207	0.101*
H10C	0.1189	0.2043	0.5876	0.101*
N1	0.26510 (15)	0.0587 (2)	0.47133 (7)	0.0689 (4)
O1	0.36592 (15)	0.0320 (3)	0.45180 (6)	0.1156 (7)
O2	0.17592 (15)	0.0466 (3)	0.44070 (7)	0.1107 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0436 (8)	0.0448 (8)	0.0577 (9)	0.0026 (6)	−0.0012 (7)	0.0014 (7)
C2	0.0443 (8)	0.0582 (10)	0.0705 (11)	−0.0013 (7)	−0.0040 (8)	0.0051 (8)
C3	0.0588 (10)	0.0554 (10)	0.0788 (12)	−0.0025 (8)	−0.0221 (9)	−0.0002 (8)
C4	0.0758 (11)	0.0482 (9)	0.0602 (10)	0.0081 (8)	−0.0150 (9)	−0.0001 (8)
C5	0.0675 (11)	0.0592 (10)	0.0576 (10)	−0.0005 (8)	0.0030 (8)	0.0056 (8)
C6	0.0527 (9)	0.0527 (9)	0.0587 (9)	−0.0081 (7)	−0.0020 (7)	0.0009 (7)
C7	0.134 (2)	0.0821 (14)	0.0653 (12)	0.0051 (14)	−0.0291 (13)	−0.0057 (10)
C8	0.0474 (8)	0.0563 (9)	0.0555 (9)	0.0029 (7)	0.0031 (7)	0.0019 (7)
C9	0.0535 (8)	0.0525 (9)	0.0512 (8)	0.0023 (7)	−0.0009 (7)	0.0009 (7)
C10	0.0500 (9)	0.0809 (12)	0.0718 (11)	0.0055 (9)	−0.0033 (8)	0.0057 (9)
N1	0.0687 (10)	0.0811 (11)	0.0570 (9)	0.0123 (8)	−0.0067 (8)	−0.0012 (8)
O1	0.0869 (11)	0.197 (2)	0.0625 (9)	0.0435 (12)	0.0054 (8)	−0.0159 (10)
O2	0.0889 (11)	0.1695 (18)	0.0737 (10)	0.0049 (11)	−0.0255 (8)	−0.0238 (10)

Geometric parameters (Å, º)

C1—C2	1.396 (2)	C7—H7A	0.9600
C1—C6	1.394 (2)	C7—H7B	0.9600
C1—C8	1.468 (2)	C7—H7C	0.9600
C2—C3	1.379 (2)	C8—C9	1.329 (2)
C2—H2	0.9300	C8—H8	0.9300
C3—C4	1.386 (3)	C9—N1	1.478 (2)
C3—H3	0.9300	C9—C10	1.488 (2)
C4—C5	1.390 (2)	C10—H10A	0.9600
C4—C7	1.504 (3)	C10—H10B	0.9600
C5—C6	1.378 (2)	C10—H10C	0.9600
C5—H5	0.9300	N1—O2	1.2094 (19)
C6—H6	0.9300	N1—O1	1.217 (2)

C2—C1—C6	117.50 (15)	H7A—C7—H7B	109.5
C2—C1—C8	118.77 (14)	C4—C7—H7C	109.5
C6—C1—C8	123.69 (14)	H7A—C7—H7C	109.5
C3—C2—C1	120.94 (16)	H7B—C7—H7C	109.5
C3—C2—H2	119.5	C9—C8—C1	128.09 (14)
C1—C2—H2	119.5	C9—C8—H8	116.0
C2—C3—C4	121.78 (16)	C1—C8—H8	116.0
C2—C3—H3	119.1	C8—C9—N1	116.07 (14)
C4—C3—H3	119.1	C8—C9—C10	129.97 (15)
C3—C4—C5	116.99 (16)	N1—C9—C10	113.95 (14)
C3—C4—C7	121.66 (18)	C9—C10—H10A	109.5
C5—C4—C7	121.35 (19)	C9—C10—H10B	109.5
C6—C5—C4	122.02 (16)	H10A—C10—H10B	109.5
C6—C5—H5	119.0	C9—C10—H10C	109.5
C4—C5—H5	119.0	H10A—C10—H10C	109.5
C5—C6—C1	120.70 (15)	H10B—C10—H10C	109.5

C5—C6—H6	119.7	O2—N1—O1	121.78 (17)
C1—C6—H6	119.7	O2—N1—C9	118.09 (16)
C4—C7—H7A	109.5	O1—N1—C9	120.13 (15)
C4—C7—H7B	109.5		

C6—C1—C2—C3	−3.5 (2)	C8—C1—C6—C5	179.77 (15)
C8—C1—C2—C3	178.93 (15)	C2—C1—C8—C9	−150.12 (16)
C1—C2—C3—C4	2.3 (3)	C6—C1—C8—C9	32.5 (3)
C2—C3—C4—C5	0.2 (2)	C1—C8—C9—N1	−176.58 (15)
C2—C3—C4—C7	179.15 (17)	C1—C8—C9—C10	4.9 (3)
C3—C4—C5—C6	−1.4 (2)	C8—C9—N1—O2	178.77 (17)
C7—C4—C5—C6	179.65 (17)	C10—C9—N1—O2	−2.5 (2)
C4—C5—C6—C1	0.1 (3)	C8—C9—N1—O1	−0.8 (3)
C2—C1—C6—C5	2.3 (2)	C10—C9—N1—O1	177.99 (18)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···O1	0.93	2.28	2.677 (2)	105
C8—H8···O1^{i}	0.93	2.55	3.369 (2)	147
C2—H2···O2^{ii}	0.93	2.66	3.551 (3)	162

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