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(E)-2-(2-Nitroprop-1-enyl)thiophene

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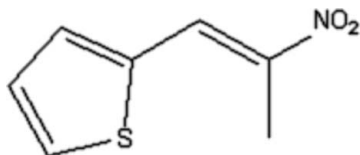
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.049; wR factor = 0.170; data-to-parameter ratio = 13.4.

The title compound, $\text{C}_7\text{H}_7\text{NO}_2\text{S}$, adopts an *E* conformation about the $\text{C}=\text{C}$ bond. The torsion angle $\text{C}=\text{C}-\text{C}-\text{C}$ is $-177.7(3)^\circ$. The crystal structure features weak intermolecular by $\text{C}-\text{H}\cdots\text{O}$ interactions.

Related literature

For the use of nitroalkenes as organic intermediates, see: Ballini & Petrini (2004); Berner *et al.* (2002); Ono (2001).



Experimental

Crystal data

 $\text{C}_7\text{H}_7\text{NO}_2\text{S}$
 $M_r = 169.20$

 Monoclinic, $P2_1/n$
 $a = 6.7545(6)$ Å

 $b = 16.6940(13)$ Å

 $c = 7.4527(4)$ Å

 $\beta = 110.640(7)^\circ$
 $V = 786.42(10)$ Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 0.36$ mm⁻¹
 $T = 296$ K

 $0.31 \times 0.18 \times 0.17$ mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.879$, $T_{\max} = 0.942$

5936 measured reflections
1362 independent reflections
971 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.170$
 $S = 1.00$
1362 reflections

102 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.39$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C6}-\text{H6}\cdots\text{O2}^i$	0.93	2.60	3.511 (5)	168

 Symmetry code: (i) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$.

Data collection: *PROCESS-AUTO* (Rigaku, 2006); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku Americas and 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 are grateful to Mr Jianming Gu for the X-ray single crystal analysis.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2274).

References

- Ballini, R. & Petrini, M. (2004). *Tetrahedron*, **60**, 1017–1047.
 Berner, O. M., Tedeschi, L. & Enders, D. (2002). *Eur. J. Org. Chem.* **12**, 1877–1894.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
 Ono, N. (2001). *The Nitro Group in Organic Synthesis*. New York: Wiley-VCH.
 Rigaku (2006). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
 Rigaku Americas and Rigaku (2007). *CrystalStructure*. Rigaku Americas, The Woodlands, Texas, USA, and Rigaku Corporation, Tokyo, Japan.
 Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.

supporting information

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(E)-2-(2-Nitroprop-1-enyl)thiophene**Zhao-Bo Li, Li-Li Shen 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 crystal structure of the title compound. The C2=C3 bond involves the *E* configuration with the C2—C3—C4—C5 torsion angle of 177.71 (3)° (Fig. 1). The atoms of the thiophene ring are coplanar. The conformation of (I) is stabilized by weak intermolecular by C6—H6···O2' interaction (Fig. 2 and Table 1).

S2. Experimental

To a solution of thiophene-2-carbaldehyde (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 an cyclohexane-EtOAc solution (10:1, v/v).

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 $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ or $1.5 U_{\text{eq}}(\text{C})$ for methyl H atoms.

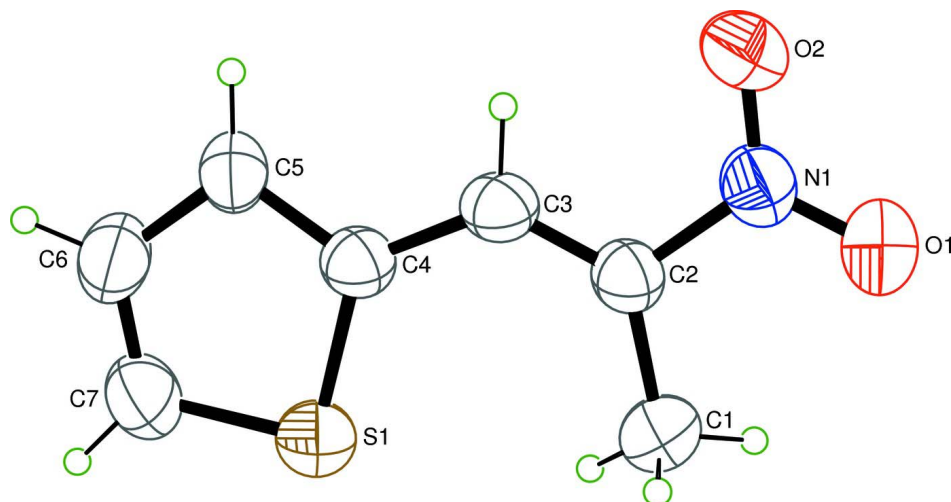


Figure 1

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

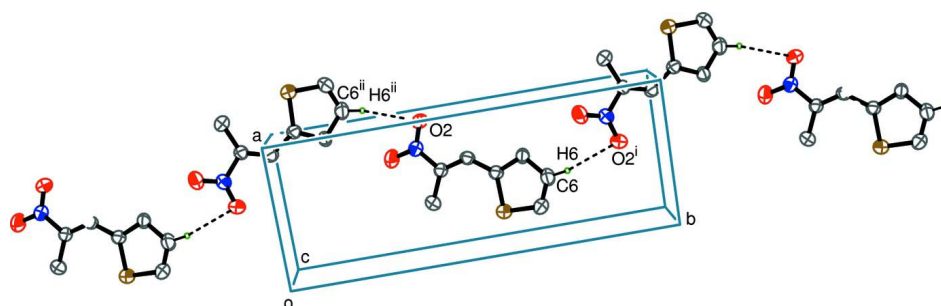


Figure 2

The view of intermolecular interaction illustrated as dash lines.

(E)-2-(2-Nitroprop-1-enyl)thiophene

Crystal data

$C_7H_7NO_2S$

$M_r = 169.20$

Monoclinic, $P2_1/n$

Hall symbol: $-P 2_1n$

$a = 6.7545 (6) \text{ \AA}$

$b = 16.6940 (13) \text{ \AA}$

$c = 7.4527 (4) \text{ \AA}$

$\beta = 110.640 (7)^\circ$

$V = 786.42 (10) \text{ \AA}^3$

$Z = 4$

$F(000) = 352$

$D_x = 1.429 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3534 reflections

$\theta = 3.2\text{--}27.4^\circ$

$\mu = 0.36 \text{ mm}^{-1}$

$T = 296 \text{ K}$

Prism, yellow

$0.31 \times 0.18 \times 0.17 \text{ mm}$

Data collection

Rigaku R-AXIS RAPID
diffractometer

Radiation source: rolling anode

Graphite monochromator

Detector resolution: $10.00 \text{ pixels mm}^{-1}$

ω scans

Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.879$, $T_{\max} = 0.942$

5936 measured reflections

1362 independent reflections
 971 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$
 $\theta_{\text{max}} = 25.0^\circ$, $\theta_{\text{min}} = 3.2^\circ$

$h = -7 \rightarrow 8$
 $k = -19 \rightarrow 19$
 $l = -8 \rightarrow 8$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.049$
 $wR(F^2) = 0.170$
 $S = 1.00$
 1362 reflections
 102 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
 $w = 1/[\sigma^2(F_o^2) + (0.0837P)^2 + 0.8184P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.39 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.33 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick,
 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.0067 (6)

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>	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.21787 (15)	0.56891 (6)	0.61453 (16)	0.0684 (5)
C4	0.4914 (5)	0.5654 (2)	0.7041 (5)	0.0496 (8)
C3	0.6192 (5)	0.4976 (2)	0.7933 (4)	0.0500 (8)
H3	0.7644	0.5067	0.8373	0.060*
N1	0.7349 (5)	0.36806 (18)	0.9203 (4)	0.0584 (8)
O2	0.9190 (4)	0.39008 (17)	0.9632 (5)	0.0775 (9)
C2	0.5624 (5)	0.4238 (2)	0.8227 (5)	0.0493 (8)
O1	0.6885 (5)	0.30107 (17)	0.9578 (5)	0.0832 (9)
C5	0.5793 (6)	0.64019 (19)	0.6781 (5)	0.0512 (8)
H5	0.7230	0.6515	0.7150	0.061*
C1	0.3450 (6)	0.3888 (2)	0.7708 (6)	0.0652 (10)
H1A	0.2975	0.3941	0.8775	0.098*
H1B	0.3487	0.3332	0.7398	0.098*
H1C	0.2496	0.4168	0.6621	0.098*
C7	0.2176 (6)	0.6653 (2)	0.5463 (6)	0.0698 (11)
H7	0.0948	0.6947	0.4869	0.084*
C6	0.4120 (6)	0.6951 (2)	0.5863 (6)	0.0675 (11)
H6	0.4365	0.7475	0.5568	0.081*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0499 (6)	0.0587 (7)	0.0895 (8)	-0.0017 (4)	0.0158 (5)	0.0006 (5)
C4	0.0447 (19)	0.051 (2)	0.0507 (19)	-0.0015 (14)	0.0131 (15)	-0.0040 (14)
C3	0.0419 (17)	0.052 (2)	0.052 (2)	-0.0039 (14)	0.0124 (15)	-0.0078 (15)
N1	0.0557 (19)	0.0507 (19)	0.0660 (19)	0.0060 (14)	0.0180 (15)	0.0000 (14)
O2	0.0474 (15)	0.0661 (18)	0.107 (2)	0.0055 (12)	0.0125 (15)	0.0016 (16)
C2	0.0468 (18)	0.0464 (19)	0.0532 (19)	0.0018 (14)	0.0157 (15)	-0.0040 (14)
O1	0.079 (2)	0.0533 (17)	0.118 (3)	0.0078 (14)	0.0349 (18)	0.0175 (16)
C5	0.056 (2)	0.0418 (18)	0.0499 (19)	0.0022 (14)	0.0116 (15)	0.0006 (14)
C1	0.054 (2)	0.056 (2)	0.082 (3)	-0.0085 (17)	0.0198 (19)	0.0007 (19)
C7	0.058 (2)	0.058 (2)	0.083 (3)	0.0100 (18)	0.012 (2)	0.002 (2)
C6	0.074 (3)	0.047 (2)	0.076 (3)	-0.0032 (18)	0.019 (2)	0.0038 (18)

Geometric parameters (\AA , $^\circ$)

S1—C7	1.688 (4)	C2—C1	1.499 (5)
S1—C4	1.730 (3)	C5—C6	1.429 (5)
C4—C5	1.425 (5)	C5—H5	0.9300
C4—C3	1.436 (5)	C1—H1A	0.9600
C3—C2	1.331 (5)	C1—H1B	0.9600
C3—H3	0.9300	C1—H1C	0.9600
N1—O1	1.220 (4)	C7—C6	1.336 (5)
N1—O2	1.226 (4)	C7—H7	0.9300
N1—C2	1.469 (4)	C6—H6	0.9300
C7—S1—C4	92.09 (18)	C4—C5—H5	125.4
C5—C4—C3	122.8 (3)	C6—C5—H5	125.4
C5—C4—S1	110.9 (2)	C2—C1—H1A	109.5
C3—C4—S1	126.3 (3)	C2—C1—H1B	109.5
C2—C3—C4	130.0 (3)	H1A—C1—H1B	109.5
C2—C3—H3	115.0	C2—C1—H1C	109.5
C4—C3—H3	115.0	H1A—C1—H1C	109.5
O1—N1—O2	122.3 (3)	H1B—C1—H1C	109.5
O1—N1—C2	118.1 (3)	C6—C7—S1	113.0 (3)
O2—N1—C2	119.6 (3)	C6—C7—H7	123.5
C3—C2—N1	116.3 (3)	S1—C7—H7	123.5
C3—C2—C1	129.2 (3)	C7—C6—C5	114.7 (4)
N1—C2—C1	114.5 (3)	C7—C6—H6	122.7
C4—C5—C6	109.3 (3)	C5—C6—H6	122.7
C7—S1—C4—C5	-0.3 (3)	O1—N1—C2—C1	-3.2 (5)
C7—S1—C4—C3	179.8 (3)	O2—N1—C2—C1	177.6 (3)
C5—C4—C3—C2	-177.7 (3)	C3—C4—C5—C6	-179.8 (3)
S1—C4—C3—C2	2.1 (6)	S1—C4—C5—C6	0.3 (4)
C4—C3—C2—N1	-179.7 (3)	C4—S1—C7—C6	0.2 (4)
C4—C3—C2—C1	0.0 (6)	S1—C7—C6—C5	-0.1 (5)

O1—N1—C2—C3	176.6 (3)	C4—C5—C6—C7	-0.2 (5)
O2—N1—C2—C3	-2.6 (5)		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
C6—H6...O2 ⁱ	0.93	2.60	3.511 (5)	168

Symmetry code: (i) $-x+3/2, y+1/2, -z+3/2$.