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(E)-2-(2-Nitroethenyl)furan

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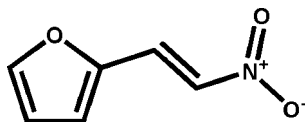
Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å;

R factor = 0.041; wR factor = 0.107; data-to-parameter ratio = 15.2.

The title compound, $\text{C}_6\text{H}_5\text{NO}_3$, was synthesized *via* condensation of furfural with nitromethane in the presence of isobutylamine. The compound crystallizes exclusively as the *E* isomer. The angle between the mean planes of the furan ring and the nitroalkenyl group is 1.3 (2)°.

Related literature

For general background, see: Wang *et al.* (2009); An *et al.* (2007); Rastogi *et al.* (2006); Rao *et al.* (2005); Negrín *et al.* (2002, 2003); Vallejos *et al.* (2005). For related structures, see: Martínez-Bescos *et al.* (2008); Novoa-de-Armas *et al.* (1997); Pomes *et al.* (1995).



Experimental

Crystal data

$\text{C}_6\text{H}_5\text{NO}_3$

$M_r = 139.11$

Monoclinic, $P2_1/n$

$a = 9.0374$ (18) Å

$b = 5.2012$ (10) Å

$c = 13.027$ (3) Å

$\beta = 97.58$ (3)°

$V = 607.0$ (2) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 0.13$ mm⁻¹

$T = 100$ K

$0.47 \times 0.17 \times 0.14$ mm

Data collection

Bruker SMART APEX
diffractometer

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 2004)

$T_{\min} = 0.916$, $T_{\max} = 0.980$

4852 measured reflections

1387 independent reflections

1317 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.107$

$S = 1.06$

1387 reflections

91 parameters

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.25$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.28$ e Å⁻³

Data collection: *SMART* (Bruker, 2001); cell refinement: *S SAINT* (Bruker, 2001); data reduction: *S SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2237).

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supplementary materials

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(*E*)-2-(2-Nitroethenyl)furan

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Comment

Among the biological properties of (nitro-alkenyl)-furan compounds our interest is focused in their antibacterial and anti-fungal activities. In spite of the importance of the structure to explain physical and chemical properties, there are not reports on the structures of the more simple compounds in this family. We start with this study a series of structural reports about them. The structure of title compound, showing *trans* or *E* configuration, is shown in Fig. 1. Ring aromaticity is extended to the alkenyl group being C1—C5 bond length, 1.430 (2), significantly shorter than a single C—C bond. Alkenyl sp^2 carbons maintain coplanarity with furan ring as shown by an angle of 1.3 (2)° between ring plane and C5—C6—N1 plane. Crystal packing does not show hydrogen bonds nor $N\cdots\pi$ intermolecular interactions (Fig. 2).

Experimental

2-(2-Nitro-ethenyl)-furan, also called G-0, was obtained by a variation of Knoevenagel's method: condensation of an aldehyde with substances containing an active α -hydrogen in the presence of a base (ammonia or amines) as catalyst. The Centro de Bioactivos Químicos (Cuba) has already patented this modified method using furfural, an aromatic compound from acid hydrolysis of sugar cane residuals (straw, sawdust, *etc.*) and nitromethane in the presence of isobutylamine. A yellow crystalline solid was obtained with purity higher than 98%, melting point 74.5°, scarcely soluble in water and very soluble in nitromethane, carbon tetrachloride, petroleum ether and ethanol.

Refinement

All H atoms were positioned geometrically and treated as riding (C—H = 0.99Å for methylene and C—H = 0.93Å otherwise). $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ of the carrier atom.

Figures

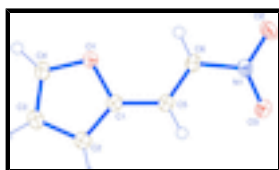


Fig. 1. ORTEP representation of the molecular structure of the title compound showing the atom labelling scheme (thermal ellipsoid probability 50%).

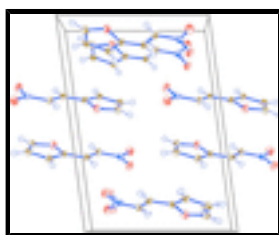


Fig. 2. Packing diagram of the title compound.

(E)-2-(2-Nitroethenyl)furan

Crystal data

$C_6H_5NO_3$	$F_{000} = 288$
$M_r = 139.11$	$D_x = 1.522 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2yn	Cell parameters from 2396 reflections
$a = 9.0374 (18) \text{ \AA}$	$\theta = 2.6\text{--}27.5^\circ$
$b = 5.2012 (10) \text{ \AA}$	$\mu = 0.13 \text{ mm}^{-1}$
$c = 13.027 (3) \text{ \AA}$	$T = 100 \text{ K}$
$\beta = 97.58 (3)^\circ$	Prism, yellow
$V = 607.0 (2) \text{ \AA}^3$	$0.47 \times 0.17 \times 0.14 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART APEX diffractometer	1387 independent reflections
Radiation source: fine-focus sealed tube	1317 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.023$
$T = 100 \text{ K}$	$\theta_{\text{max}} = 27.5^\circ$
1700 ω scan frames (0.3° , 10)	$\theta_{\text{min}} = 2.6^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$h = -11 \rightarrow 11$
$T_{\text{min}} = 0.916$, $T_{\text{max}} = 0.980$	$k = -6 \rightarrow 6$
4852 measured reflections	$l = -16 \rightarrow 12$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 0.266P]$
$R[F^2 > 2\sigma(F^2)] = 0.041$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.107$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.25 \text{ e \AA}^{-3}$
1387 reflections	$\Delta\rho_{\text{min}} = -0.28 \text{ e \AA}^{-3}$
91 parameters	Extinction correction: none

Special details

Experimental. Refinement of F^2 against unique set of 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 $F^2 > 2\sigma(F^2)$ 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.

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 unique set of 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.67495 (10)	0.19614 (17)	0.08110 (7)	0.0206 (2)
O2	0.15283 (11)	0.49888 (19)	0.10417 (8)	0.0290 (3)
O3	0.16242 (10)	0.13012 (19)	0.18291 (7)	0.0254 (3)
N1	0.21943 (12)	0.3007 (2)	0.13567 (8)	0.0205 (3)
C1	0.59854 (14)	0.0119 (2)	0.12894 (9)	0.0182 (3)
C2	0.68866 (14)	-0.1941 (2)	0.15531 (9)	0.0202 (3)
H2	0.6627	-0.3465	0.1890	0.024*
C3	0.82904 (14)	-0.1370 (3)	0.12270 (10)	0.0221 (3)
H3	0.9152	-0.2435	0.1300	0.027*
C4	0.81535 (14)	0.0996 (3)	0.07909 (10)	0.0227 (3)
H4	0.8930	0.1870	0.0508	0.027*
C5	0.44755 (13)	0.0618 (2)	0.14455 (9)	0.0185 (3)
H5	0.3974	-0.0667	0.1787	0.022*
C6	0.37162 (14)	0.2755 (3)	0.11477 (10)	0.0197 (3)
H6	0.4172	0.4090	0.0803	0.024*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0179 (4)	0.0200 (5)	0.0243 (5)	0.0009 (3)	0.0046 (3)	0.0019 (3)
O2	0.0269 (5)	0.0301 (5)	0.0303 (5)	0.0112 (4)	0.0053 (4)	0.0047 (4)
O3	0.0206 (5)	0.0269 (5)	0.0299 (5)	-0.0014 (4)	0.0074 (4)	0.0019 (4)
N1	0.0189 (5)	0.0244 (6)	0.0182 (5)	0.0026 (4)	0.0023 (4)	-0.0017 (4)
C1	0.0199 (6)	0.0185 (6)	0.0163 (6)	-0.0013 (4)	0.0027 (4)	-0.0017 (4)
C2	0.0221 (6)	0.0199 (6)	0.0186 (6)	0.0006 (5)	0.0019 (5)	-0.0010 (5)
C3	0.0201 (6)	0.0256 (6)	0.0204 (6)	0.0042 (5)	0.0016 (5)	-0.0025 (5)
C4	0.0164 (6)	0.0279 (7)	0.0242 (6)	0.0007 (5)	0.0040 (5)	-0.0011 (5)
C5	0.0185 (6)	0.0212 (6)	0.0159 (6)	-0.0021 (5)	0.0028 (4)	-0.0023 (4)
C6	0.0170 (6)	0.0236 (6)	0.0194 (6)	0.0002 (5)	0.0052 (4)	-0.0013 (5)

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

O1—C4	1.3680 (15)	C2—H2	0.9500
O1—C1	1.3772 (15)	C3—C4	1.3544 (19)
O2—N1	1.2361 (14)	C3—H3	0.9500

supplementary materials

O3—N1	1.2309 (15)	C4—H4	0.9500
N1—C6	1.4428 (16)	C5—C6	1.3366 (18)
C1—C2	1.3623 (17)	C5—H5	0.9500
C1—C5	1.4296 (17)	C6—H6	0.9500
C2—C3	1.4214 (18)		
C4—O1—C1	105.97 (10)	C4—C3—H3	126.9
O3—N1—O2	123.33 (11)	C2—C3—H3	126.9
O3—N1—C6	120.08 (11)	C3—C4—O1	111.04 (12)
O2—N1—C6	116.59 (11)	C3—C4—H4	124.5
C2—C1—O1	110.03 (11)	O1—C4—H4	124.5
C2—C1—C5	131.07 (12)	C6—C5—C1	124.94 (12)
O1—C1—C5	118.89 (11)	C6—C5—H5	117.5
C1—C2—C3	106.73 (11)	C1—C5—H5	117.5
C1—C2—H2	126.6	C5—C6—N1	119.11 (12)
C3—C2—H2	126.6	C5—C6—H6	120.4
C4—C3—C2	106.23 (11)	N1—C6—H6	120.4
C4—O1—C1—C2	-0.39 (13)	C1—O1—C4—C3	0.54 (14)
C4—O1—C1—C5	178.58 (10)	C2—C1—C5—C6	179.68 (13)
O1—C1—C2—C3	0.12 (14)	O1—C1—C5—C6	0.96 (18)
C5—C1—C2—C3	-178.69 (12)	C1—C5—C6—N1	-179.91 (11)
C1—C2—C3—C4	0.21 (14)	O3—N1—C6—C5	2.03 (17)
C2—C3—C4—O1	-0.47 (14)	O2—N1—C6—C5	-178.15 (11)

Fig. 1

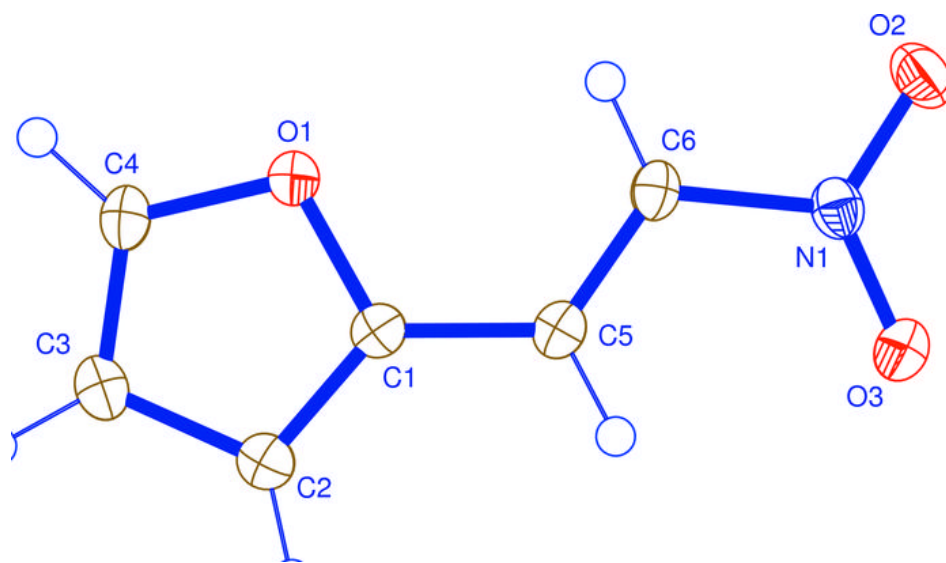


Fig. 2

