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# (2*E*)-3-[(1*E*)-2-(5-Nitroisoxazol-3-yl)vinyloxy]-prop-2-enal

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**Key indicators**

Single-crystal X-ray study
*T* = 294 K
Mean σ(C–C) = 0.003 Å
*R* factor = 0.036
*wR* factor = 0.101
Data-to-parameter ratio = 9.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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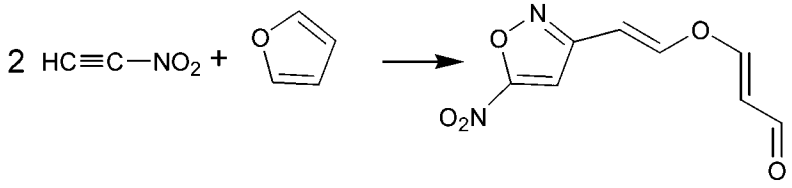
The title compound, (2*E*)-3-[(1*E*)-2-(5-nitroisoxazol-3-yl)-vinyloxy]prop-2-enal, $C_8H_6N_2O_5$, was an unexpected product of the direct reaction of furan and nitroacetylene. It crystallizes in the monoclinic space group $P2_1/c$, and is an unsaturated compound with a density of 1.507 g cm$^{-3}$. As a result of cooperative C—H···O hydrogen bonding, the title compound forms centrosymmetric planar dimers, which pack in stacks of almost-planar parallel layers.

## Comment

Nitroacetylene was recently synthesized by Zhang *et al.* (2002) and was found to be relatively stable in solution ($CH_2Cl_2$ or $CHCl_3$; 0.1 to 1.0 *M*) for a few days at room temperature, and indefinitely at 195 K. This very active reagent acts as a dienophile in a simple (1:1) Diels–Alder condensation with cyclopentadiene, but reacts to give a more complex product with furan. This product, the title compound, (I), was supplied in the form of a few very small crystals, with an uncertain empirical formula; thus, it was treated as an unknown containing C, H, N, and O atoms.



(I)

A well shaped, but small (0.02 × 0.12 × 0.16 mm), crystal was used to collect data on a CCD diffractometer with a rotating anode Cu *Kα* source. As a result of the small crystal volume, the scattering disappeared into the background at a *d* spacing of 0.9 Å; the mean $I/\sigma$ was only 1.10 for 183 reflections with $0.9 < d < 0.95$ Å. Analysis by direct methods revealed the framework immediately and least-squares refinements were used to successively test the identity of each ring atom, by treating the atom occupancy as a free variable. All H atoms did show up clearly as this refinement proceeded, facilitating the elemental assignments. The final result coincided exactly in stoichiometry to the sum of two equivalents of nitroacetylene adding to one furan molecule. The appearance of a ring other than furan in the product was surprising. A suggested mechanism, as well as supportive NMR and mass spectrometric evidence, has been reported by Zhang *et al.* (2002). A search of the Cambridge Structural Database (Allen & Kennard, 1993; Version 5.23 of April 2002, using *Conquest* 1.4) revealed only one nitro-substituted isoxazole, 3,5-dinitroisoxazole (Cromer *et al.*, 1987).

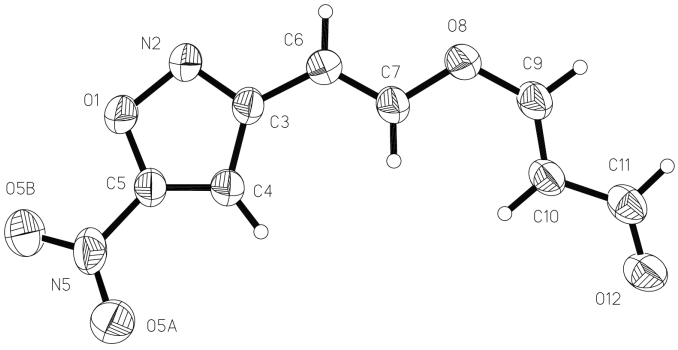



**Figure 1**
A view of the title compound, with 50% probability ellipsoids.

There are three short intermolecular contacts (listed in Table I) between the terminal carbonyl, O12, and three H atoms H4, H7, and H10 on the inside of the curved polyene chain (of a symmetry-related neighbor). These cooperative hydrogen bonds link the molecules into weakly bound dimers. The planar dimers pack in stacks, forming a crystal in which all of the molecules are nearly parallel to one another. There are slight ripples, or waves, in the semi-planar sheets. The sheets pass through the unit cell in a diagonal direction, parallel to the $(10\overline{4})$ crystal planes.

## Experimental

Dr Mao-Xi Zhang of the Chemistry Department, University of Chicago, supplied crystals of the title compound.

*Crystal data*

$C_8H_6N_2O_5$
$M_r$ = 210.15
Monoclinic, $P2_1/c$
$a$ = 3.9575 (2) Å
$b$ = 12.3228 (4) Å
$c$ = 19.0970 (7) Å
$\beta$ = 95.849 (2)°
$V$ = 926.46 (7) Å$^3$
$Z$ = 4
$D_x$ = 1.507 Mg m$^{-3}$
Cu $K\alpha$ radiation
Cell parameters from 2436 reflections
$\theta$ = 4.3–57.3°
$\mu$ = 1.12 mm$^{-1}$
$T$ = 294 (2) K
Lath, colorless
0.16 × 0.12 × 0.02 mm

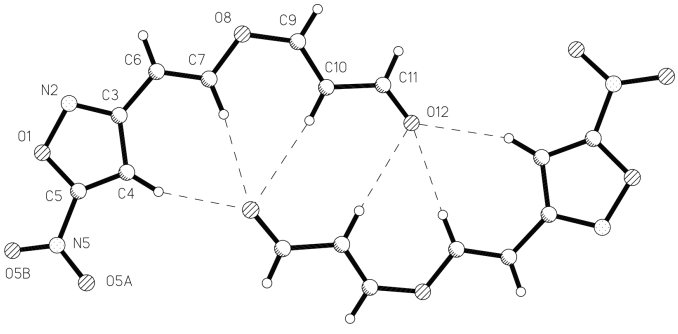



**Figure 2**
A view of the centrosymmetric planar dimer formed by the title compound, using six cooperative hydrogen bonds.

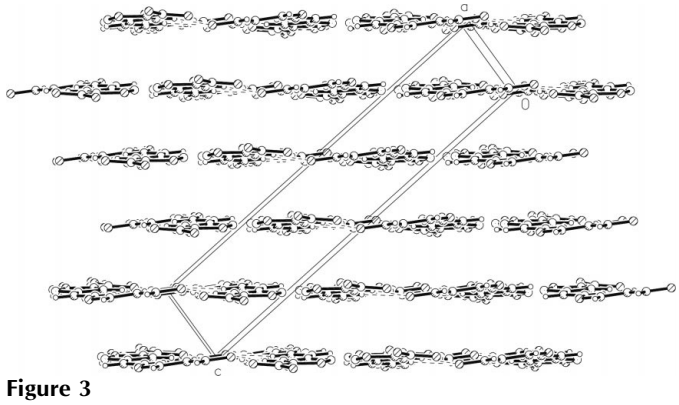



**Figure 3**
A packing diagram of the molecule. The planar dimers pack in stacks nearly parallel to one another.

*Data collection*

Bruker SMART CCD area-detector diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min}$ = 0.86, $T_{max}$ = 0.98
6207 measured reflections
1323 independent reflections
978 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.045
$\theta_{max}$ = 58.9°
$h = -4 \rightarrow 4$
$k = -13 \rightarrow 13$
$l = -20 \rightarrow 21$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.101
$S$ = 1.02
1323 reflections
137 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0354P)^2 + 0.1275P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.14 e Å$^{-3}$
Extinction correction: *SHELXL*
Extinction coefficient: 0.0083 (10)

**Table 1**
Hydrogen-bonding geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C10—H10···O12$^{i}$ | 0.93 | 2.62 | 3.532 (3) | 168 |
| C7—H7···O12$^{i}$ | 0.93 | 2.44 | 3.362 (2) | 171 |
| C4—H4···O12$^{i}$ | 0.93 | 2.40 | 3.282 (3) | 159 |

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001) and *SHELXTL* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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