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4-(2-Nitrovinyl)-2,3,6,7-tetrahydrobenzo-[1,2-*b*;4,5-*b*′]difuran

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.102
Data-to-parameter ratio = 15.5

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

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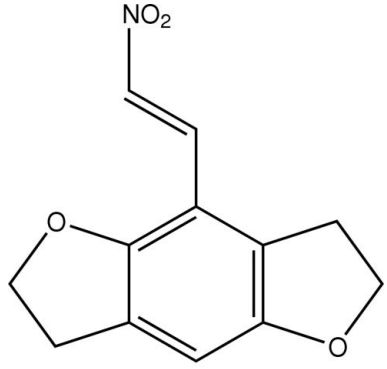
In the structure of the title compound, $C_{12}H_{11}NO_4$, the two furan rings, one to a greater degree than the other, adopt envelope conformations and are twisted slightly relative to the benzene ring. The olefinic bond displays a *trans* configuration.

Comment

Dihydrofurans are an important class of pharmacophore that are observed in many therapeutic agents. They are versatile intermediates for the synthesis of complex natural products (Dean, 1982). All the bond lengths and bond angles of the title compound lie within their expected ranges, and are comparable to those in a related compound (Gu *et al.*, 2005). The two dihydrofuran rings adopt envelope conformations and, excluding the C atoms forming the flaps of the envelopes (C3 and C8), are essentially planar. The flap atoms C3 and C8 deviate from the mean planes through the other four atoms by 0.089 (4) and 0.273 (4) Å, respectively. These two mean planes (C4—C5—C2—O1 and C9—C10—C7—O2) are twisted slightly out of the benzene ring plane, forming dihedral angles of 1.60 (11) and 2.04 (9)° respectively. The olefinic bond in the molecule diaplays a *trans* configuration and is coplanar with the benzene ring.



(I)

Experimental

4-Formyl-2,3,6,7-tetrahydrobenzo[1,2-*b*;4,5-*b*′]difuran (3.2 g, 17 mmol) and ammonium acetate (1.3 g,17 mmol) were dissolved in nitromethane (20 ml) under an N_2 atmosphere. The mixture was refluxed for 1 h and the nitromethane was then removed under reduced pressure. The solid residue was extracted with CH_2Cl_2 and water, dried with anhydrous $MgSO_4$, and recrystallized from methanol to obtain 3.4 g (yield 90%) of the title compound (Monte *et al.*, 1996). It was further recrystallized from ethanol, giving red crystals suitable for X-ray diffraction.

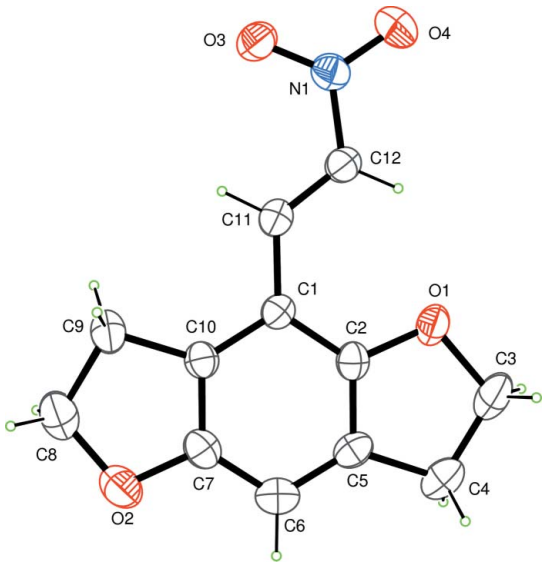


Figure 1
A view of (1). Displacement ellipsoids are drawn at the 40% probability level and H atoms are shown as small circles of arbitrary radii.

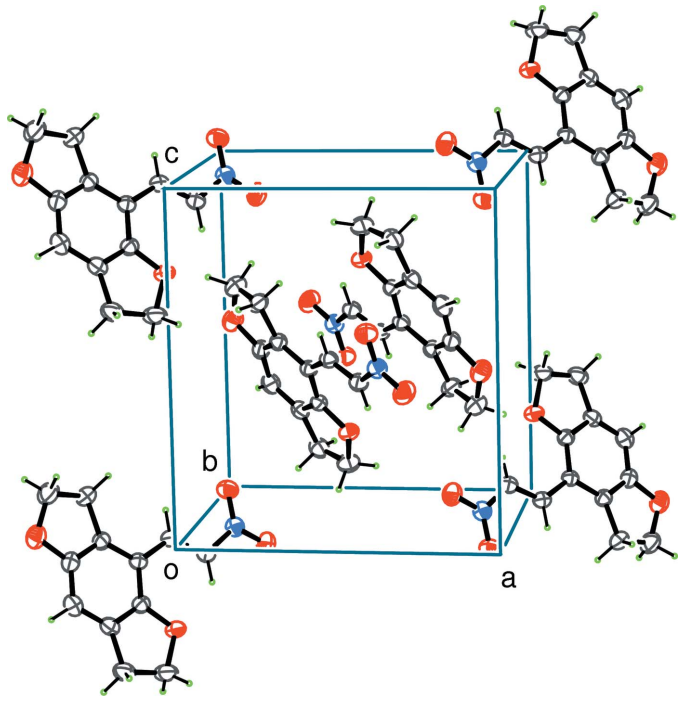


Figure 2
The molecular packing of (1).

Crystal data

$C_{12}H_{11}NO_4$
M_r = 233.22
Monoclinic, $P2_1/n$
a = 10.220 (9) Å
b = 9.204 (6) Å
c = 11.386 (7) Å
β = 90.08 (3)°
V = 1071.0 (13) Å^3
Z = 4
D_x = 1.446 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5880 reflections
θ = 3.5–27.6°
μ = 0.11 mm^{-1}
T = 296 (1) K
Prism, red
0.30 × 0.20 × 0.10 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: none
9923 measured reflections
2396 independent reflections
949 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.048
θ_{max} = 27.5°
$h = -13 \rightarrow 13$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.102
S = 1.00
2396 reflections
155 parameters
H-atom parameters constrained
$w = 1/[0.0001F_o^2 + 0.92\sigma(F_o^2)]/(4F_o^2)$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 161 (15)

Table 1
Selected bond lengths (Å).

O1—C2	1.382 (2)	O2—C8	1.450 (3)
O1—C3	1.471 (2)	N1—C12	1.444 (2)
O2—C7	1.378 (2)	C1—C2	1.405 (2)

All H atoms were placed in idealized positions (C—H = 0.93–0.97 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure*.

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