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5-Bromo-2-(2-nitroethenyl)furan

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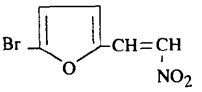
Abstract

The five-membered ring of the title compound, $C_6H_4BrNO_3$, has normal geometry and there are no unusual intramolecular distances or angles. The O atoms of the nitro group interact with the C—H groups of a neighboring molecule with H···O distances of 2.46 (1) and 2.56 (1) Å.

Comment

The structure determination of 5-bromo-2-(2-nitroethenyl)furan, (I), was undertaken as part of a molecular-modeling investigation. The crystals were obtained by slow recrystallization from ethanol. Details of the synthetic work will be published elsewhere (Castañedo & Estrada, 1995).



(I)

The structure and properties of the title compound are of great interest because of its proven pharmacological activity (Castañeda, 1993). A summary of the bond distances and angles, and intermolecular contacts shorter than the sum of the van der Waals radii (C 1.70, O 1.40 Å; Pauling, 1960), are listed in Table 2.

The displacement ellipsoids with the atomic numbering (*SHELXTL-Plus*; Sheldrick, 1991) are shown in Fig. 1. As shown in Fig. 2, the O atoms of the nitro group interact with the C—H groups of a neighboring molecule [C(4)—H(4)···O(2) and C(6)—H(6)···O(3) with H···O distances of 2.56 (1) and 2.46 (1) Å, respectively].

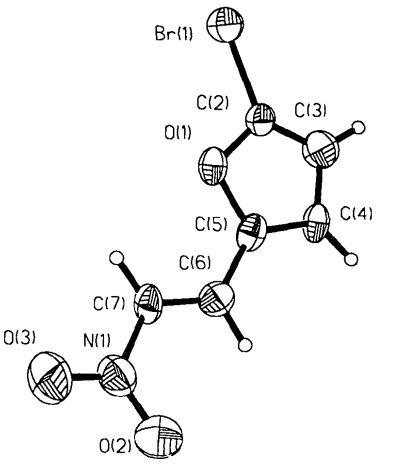


Fig. 1. Displacement ellipsoid plot (*SHELXTL-Plus*; Sheldrick, 1991) of the title compound. Ellipsoids are scaled to enclose 50% probability and H atoms are represented as spheres of arbitrary radii.

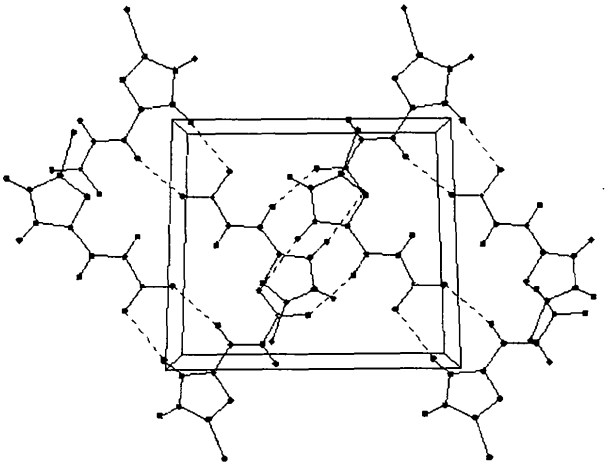


Fig. 2. The packing of the molecules in the unit cell. Hydrogen bonds are represented by dashed lines.

Experimental

Crystal data

$C_6H_4BrNO_3$
M_r = 218.01
Monoclinic
$P2_1/c$
a = 7.370 (1) Å
b = 10.919 (2) Å
c = 9.887 (1) Å
β = 108.45 (1)°

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 23 reflections
θ = 2.86–25°
μ = 5.3 mm^{-1}
T = 293 K
Prismatic

V = 754.7 (2) Å^3
Z = 4
D_x = 1.920 Mg m^{-3}
0.20 × 0.15 × 0.12 mm
Colorless

Data collection

Siemens *P*3/PC diffractometer
$2\theta/\theta$ scans
Absorption correction: refined from ΔF
T_{min} = 0.25, T_{max} = 0.60
1411 measured reflections
1305 independent reflections
776 observed reflections [$I > 2\sigma(I)$]
θ_{max} = 25°
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 11$
3 standard reflections monitored every 50 reflections
intensity decay: <3%

Refinement

Refinement on F
R = 0.05
wR = 0.06
S = 1.61
776 reflections
101 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.001600F^2]$
$(\Delta/\sigma)_{max}$ = 0.0363
$\Delta\rho_{max}$ = 0.599 e Å^{-3}
$\Delta\rho_{min}$ = −0.779 e Å^{-3}
Extinction correction: Zachariasen (1963)
Extinction coefficient: 3 (2) × 10^{-7}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Br(1)	0.8976 (2)	0.3368 (1)	0.0503 (1)	0.048 (1)
O(1)	1.1555 (11)	0.3081 (7)	0.3156 (8)	0.036 (3)
C(2)	1.0458 (15)	0.3953 (12)	0.2292 (12)	0.038 (4)
C(3)	1.0637 (20)	0.5048 (15)	0.2852 (13)	0.054 (5)
C(4)	1.1973 (19)	0.4903 (11)	0.4237 (14)	0.048 (5)
C(5)	1.2476 (17)	0.3711 (11)	0.4402 (11)	0.035 (4)
C(6)	1.3718 (17)	0.3032 (13)	0.5598 (12)	0.041 (5)
C(7)	1.4091 (18)	0.1860 (10)	0.5643 (12)	0.037 (4)
N(1)	1.5281 (16)	0.1284 (12)	0.6882 (12)	0.050 (4)
O(2)	1.6078 (18)	0.1950 (12)	0.7934 (10)	0.077 (5)
O(3)	1.5475 (17)	0.0175 (11)	0.6897 (10)	0.074 (5)

Table 2. *Selected geometric parameters* (Å, °)

Br(1)—C(2)	1.87 (1)	O(1)—C(2)	1.36 (1)
O(1)—C(5)	1.39 (1)	C(2)—C(3)	1.30 (2)
C(3)—C(4)	1.42 (2)	C(4)—C(5)	1.35 (2)
C(5)—C(6)	1.45 (2)	C(6)—C(7)	1.30 (2)
C(7)—N(1)	1.41 (2)	N(1)—O(2)	1.25 (2)
N(1)—O(3)	1.22 (2)		
H(4)···O(2)	2.56 (1)	H(6)···O(3)	2.46 (1)
C(2)—O(1)—C(5)	103.7 (9)	Br(1)—C(2)—O(1)	114.1 (9)
Br(1)—C(2)—C(3)	131.5 (9)	O(1)—C(2)—C(3)	114.4 (9)
C(2)—C(3)—C(4)	104.7 (12)	C(3)—C(4)—C(5)	107.7 (11)
O(1)—C(5)—C(4)	109.4 (9)	O(1)—C(5)—C(6)	118.5 (10)
C(4)—C(5)—C(6)	132.1 (11)	C(5)—C(6)—C(7)	126.9 (11)
C(6)—C(7)—N(1)	122.2 (10)	C(7)—N(1)—O(2)	117.5 (11)
C(7)—N(1)—O(3)	119.5 (10)	O(2)—N(1)—O(3)	123.0 (11)

Data collection: *XSCANS* (Siemens, 1992). Cell refinement: *SHELXTL-Plus* (Sheldrick, 1991) and *SHELXL*93 (Sheldrick, 1993). Data reduction, structure solution (direct methods) and software used to prepare material for publication: *SHELXTL-Plus*. All computations were carried out with an IBM 486 DX computer.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: CR1140). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Adducts from the Pyrolysis of Cellulose: (1α,2β,6β,7α)-3,8-Dioxatricyclo[5.3.1.1^{2,6}]-dodeca-4,9-diene-11,12-dione and (1*R*)-(1α,2β,3α,6α,8β,9α)-5,13,14-Trioxatetracyclo[7.3.1.1^{3,6}.0^{2,8}]tetradec-10-ene-7,12-dione

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Abstract

The compounds (1α,2β,6β,7α)-3,8-dioxatricyclo-[5.3.1.1^{2,6}]dodeca-4,9-diene-11,12-dione, $C_{10}H_8O_4$, (9), and (1*R*)-(1α,2β,3α,6α,8β,9α)-5,13,14-trioxatetracyclo-[7.3.1.1^{3,6}.0^{2,8}]tetradeca-10-ene-7,12-dione, $C_{11}H_{10}O_5$, (17), were obtained during studies of the products of