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## 2-(1,3-Benzoxazol-2-yl)-3-(5-diethylamino-2-furyl)propenenitrile

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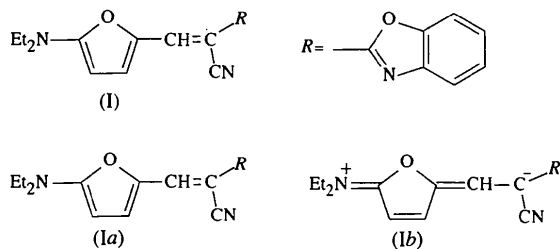
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### Abstract

The title compound, C<sub>18</sub>H<sub>17</sub>N<sub>3</sub>O<sub>2</sub>, belongs to a series of large conjugated molecules with potential use as organic dyes. As indicated by the planarity of the molecule and the pattern of bond lengths and angles, a considerable amount of  $\pi$ -electron density is transferred from the aminofuran portion of the molecule to the cyano and benzoxazolyl groups. This fact is supported by the packing of the molecules in the crystal.

### Comment

As part of our study of the relationships between molecular and electronic structures and absorption properties of large delocalized molecules as potential organic dyes, we have prepared a series of 1-(2-furyl)-2-cyanoethylenes. These compounds can be described as polarized molecules with an absorption in the near-ultraviolet and visible regions due to delocalization of  $\pi$  electrons; they exhibit pronounced colour changes depending on the nature of the substituents attached to the ethylene C atom and the furan ring. Consequently, it is of interest to examine the extent of conjugation in these molecules by the combined use of theoretical and experimental methods. We report here the crystal structure of the diethylaminobenzoxazolyl derivative (I).



As expected, the furan and benzoxazole ring systems are planar to within the limits of experimental error ( $\chi^2 = 5.99$  and 12.60, respectively). As shown from

the exocyclic torsion angles (Table 1), the molecule as a whole is also approximately planar. The N3 atom is  $sp^2$  hybridized as evidenced by the sum of the valence angles around this atom [ $360.0(4)^\circ$ ], with the lone-pair electrons available for  $\pi$  bonding. The bond distances C8—C10 [1.370(5) Å] and C10—C11 [1.371(5) Å] are the same, the C—C bonds within the furan ring are equivalent, and the C14—N3 bond length of 1.324(4) Å is intermediate between that of a pure single and double bond (Burke-Laing & Laing, 1976). These facts indicate that the neutral, (Ia), and zwitterionic, (Ib), forms are equal contributors to the ( $\pi$ ) electronic structure of the molecule. The negative charge, formally written in (Ib) on the C8 atom, is most likely transferred further and spread over the cyano and 2-benzoxazolyl groups. This is seen in the C8—C9 and C8—C1 bond lengths [1.407(5) and 1.411(5) Å, respectively] which are shorter than the values expected for pure  $C_{sp^2}$ — $C_{sp}$  and  $C_{sp^2}$ — $C_{sp^2}$  single bonds (Hummel & Procher, 1986; Shmueli, Shanan-Atidi, Horwitz & Shvo, 1973).

The crystal packing lends additional support to the distribution of  $\pi$  density in the molecule. Molecules related by the translation along the *a* axis stack in such a way that the furan ring superimposes the five-membered ring of the benzoxazole moiety. As no suitable hydrogen donors are available, the stacks are packed by van der Waals interactions.

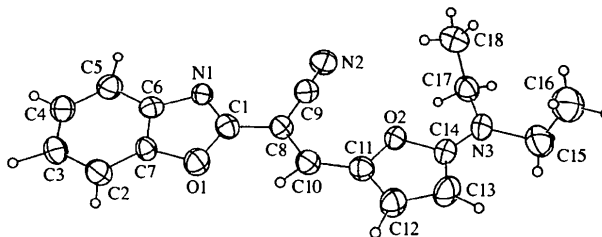


Fig. 1. A view (ORTEP; Johnson, 1976) of the title molecule showing the labelling of the non-H atoms. Displacement ellipsoids are drawn at 50% probability levels and H atoms are drawn as small circles of arbitrary radii.

### Experimental

A mixture of 2-benzoxazolylacetonitrile (0.79 g, 5 mmol), 5-diethylamino-2-furancarbaldehyde (0.83 g, 5 mmol) and glycine (0.2 g, 2.7 mmol) was heated in ethanol (60 ml) for 4 h at boiling temperature. After cooling, glycine was filtered off and washed with chloroform (15 ml). The combined organic solutions were purified with charcoal and the solvents distilled under reduced pressure. The crude product was crystallized from ethanol (yield 1.2 g, 77%).

### Crystal data

C<sub>18</sub>H<sub>17</sub>N<sub>3</sub>O<sub>2</sub>  
 $M_r = 307.35$

Mo  $K\alpha$  radiation  
 $\lambda = 0.71073$  Å

## Monoclinic

 $P2_1/c$  $a = 6.527 (1) \text{ \AA}$  $b = 17.257 (3) \text{ \AA}$  $c = 13.990 (3) \text{ \AA}$  $\beta = 93.58 (3)^\circ$  $V = 1572.7 (5) \text{ \AA}^3$  $Z = 4$  $D_x = 1.298 \text{ Mg m}^{-3}$  $D_m = 1.30 (1) \text{ Mg m}^{-3}$  $D_m$  measured by flotation in bromoform/cyclohexane

## Data collection

Syntex  $P2_1$  diffractometer $\theta/2\theta$  scans

Absorption correction: none

3757 measured reflections

3457 independent reflections

1155 reflections with

 $I > 2\sigma(I)$  $R_{\text{int}} = 0.033$ 

Cell parameters from 15 reflections

 $\theta = 13\text{--}22^\circ$  $\mu = 0.087 \text{ mm}^{-1}$  $T = 293 (2) \text{ K}$ 

Prism

 $0.40 \times 0.25 \times 0.20 \text{ mm}$ 

Dark green

 $\theta_{\text{max}} = 27.59^\circ$  $h = 0 \rightarrow 7$  $k = 0 \rightarrow 22$  $l = -18 \rightarrow 18$ 

2 standard reflections

frequency: 100 min

intensity decay: none

## Refinement

Refinement on  $F^2$  $R[F^2 > 2\sigma(F^2)] = 0.063$  $wR(F^2) = 0.140$  $S = 1.309$ 

3457 reflections

209 parameters

H atoms not refined

 $w = 1/[\sigma^2(F_o^2) + (0.1145P)^2]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\text{max}} = 0.002$  $\Delta\rho_{\text{max}} = 0.388 \text{ e \AA}^{-3}$  $\Delta\rho_{\text{min}} = -0.193 \text{ e \AA}^{-3}$ 

Extinction correction:

*SHELXL93* (Sheldrick, 1993)

Extinction coefficient:

0.004 (3)

Scattering factors from

*International Tables for Crystallography* (Vol. C)

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1099). Services for accessing these data are described at the back of the journal.

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*Acta Cryst.* (1997). **C53**, 1359–1362*p*-Iodobenzaldehyde

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Table 1. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

O1—C1	1.339 (4)	C8—C10	1.370 (5)
O1—C7	1.390 (4)	C8—C9	1.407 (5)
O2—C14	1.340 (4)	C10—C11	1.371 (5)
O2—C11	1.374 (4)	C11—C12	1.377 (5)
N1—C1	1.301 (4)	C12—C13	1.369 (5)
N3—C14	1.324 (4)	C13—C14	1.372 (5)
C1—C8	1.411 (5)		
C1—O1—C7	104.6 (3)	O1—C1—C8	120.9 (3)
C14—O2—C11	107.3 (3)	C10—C8—C9	123.3 (3)
C1—N1—C6	104.7 (3)	C10—C8—C1	121.9 (3)
C14—N3—C17	121.0 (3)	C9—C8—C1	114.7 (3)
C14—N3—C15	120.1 (4)	C8—C10—C11	130.1 (3)
C17—N3—C15	118.9 (4)	C10—C11—O2	119.7 (3)
N1—C1—O1	114.5 (3)	C10—C11—C12	132.4 (4)
N1—C1—C8	124.6 (4)	O2—C11—C12	107.9 (3)
N1—C1—C8—C10	175.7 (3)	C8—C10—C11—O2	−3.9 (6)
N1—C1—C8—C9	−3.1 (5)	C15—N3—C14—O2	−176.6 (3)
C1—C8—C10—C11	−179.2 (4)		

H atoms were located from a difference Fourier map but were not refined.  $U_{\text{iso}}$  values were set to  $1.2U_{\text{eq}}$  of the parent atom.

Data collection: Syntex  $P2_1$  software. Cell refinement: Syntex  $P2_1$  software. Data reduction: *XP21* (Pavelčík, 1987). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

## Abstract

The molecular structure of the title compound,  $\text{C}_7\text{H}_5\text{IO}$ , is normal. There are two independent molecules in the asymmetric unit. Both kinds of molecules pack in similar two-dimensional sheets, in which the most striking feature is short distances [3.068 (4) and 3.074 (4)  $\text{\AA}$ ] between the O and I atoms in adjacent molecules, both of which can be thought of as an interaction between the Lewis base,  $-\text{CHO}$ , and the Lewis acid, I.

## Comment

Aromatic nitroso compounds usually crystallize as dimers with N—N bonds although they are monomeric in solution. *p*-Iodonitrosobenzene is unusual in that it is monomeric in the solid (Webster, 1956). This appears to be the result of an intermolecular  $\text{NO} \cdots \text{I}$  interaction that is comparable in energy to the  $\text{NO}—\text{NO}$  dimerization. We have determined the structure of the title compound, (I), to examine whether a similar  $\text{CO} \cdots \text{I}$  inter-