ORGANIC COMPOUNDS

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A *Z* Isomer of 5-Nitrofuran-2-aldoxime

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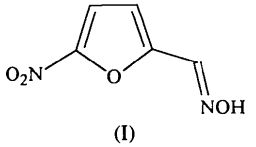
Abstract

The molecule of the title compound ($C_5H_4N_2O_4$) is practically planar; the dihedral angles between the ring plane and the planes of the nitro group and the aldoxime group are 5.0 (3) and 4.4 (3)°, respectively. Intermolecular O4—H4···N2 hydrogen bonds, with O—H = 0.96 (5), H···N = 1.89 (5), N···O = 2.827 (5) Å and O—H···N = 164 (1)°, connect molecules into infinite chains along the *b* axis.

Comment

The title compound, (I), has been found to exhibit large molecular hyperpolarizability and second-harmonic generation (SHG) efficiency (Matsuoka *et al.*, 1991; Chemla & Zyss, 1987). In the preceding paper the results of an X-ray structure analysis of the *E* isomer of (I) are reported (Olszak, Peeters, Blaton & De Ranter, 1995).



(I)

We found that at vacuum sublimation (10^{-2} mm of Hg), needle-like crystals with m.p. 391–393 K were generated. The latter turned out to be the *Z* isomer of (I). The conformation of the molecule studied is analogous to that of the corresponding thiophene derivative (Matsuoka *et al.*, 1991). The geometric data of the molecule (Fig. 1) are close to usual values (Allen *et al.*, 1987). The molecule is almost planar; the angles of rotation of the nitro and aldoxime groups around the C4—N1 and C1—C5 bonds, respectively, are 5.0 (3) and 4.4 (3)°, respectively. In the crystal structure the molecules are connected by O4—H4···N2($2 - x$, $\frac{1}{2} + y$, $1 - z$) hydrogen bonds [N···O 2.827 (3) Å] into infinite chains along the *b* axis (Fig. 2).

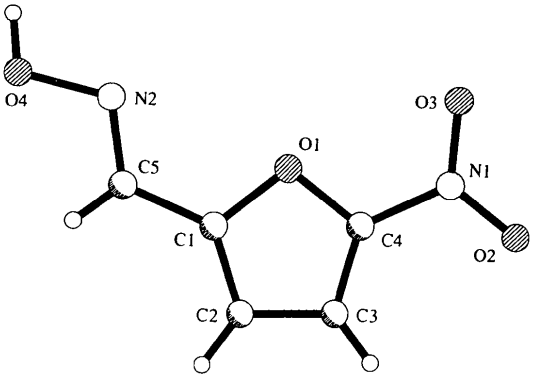


Fig. 1. View of the molecule with atom-numbering scheme.

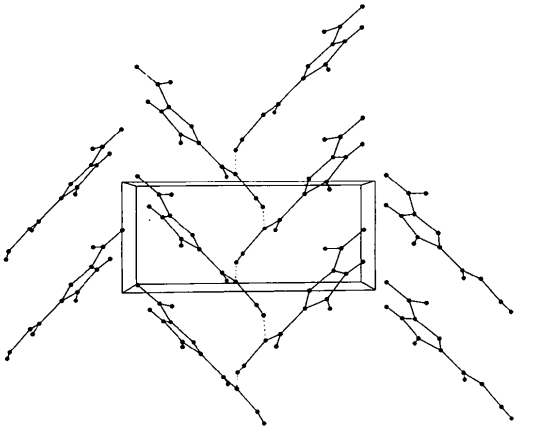


Fig. 2. Projection of the structure onto the (100) plane.

Experimental

Crystal data

$C_5H_4N_2O_4$
M_r = 156.1
Monoclinic
$P2_1$
a = 6.466 (3) Å
b = 4.634 (2) Å
c = 10.755 (5) Å
β = 94.74 (4)°
V = 321.2 (5) Å^3
Z = 2
D_x = 1.61 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 12 reflections
θ = 10–12.5°
μ = 0.134 mm^{-1}
T = 293 K
Prism
0.30 × 0.25 × 0.10 mm
Colourless

Data collection

Syntex $P\bar{1}$ diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1149 measured reflections
1089 independent reflections
567 observed reflections [$I \geq 2\sigma(I)$]
R_{int} = 0.017
θ_{max} = 25°
$h = -7 \rightarrow 7$
$k = 0 \rightarrow 5$
$l = 0 \rightarrow 12$
3 standard reflections monitored every 100 reflections
intensity decay: 2%

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + 0.0425F_o^2]$
$R(F) = 0.030$	$(\Delta/\sigma)_{max} = 0.03$
$wR(F^2) = 0.080$	$\Delta\rho_{max} = 0.12$ e Å^{-3}
$S = 1.13$	$\Delta\rho_{min} = -0.14$ e Å^{-3}
567 reflections	Extinction correction: none
115 parameters	Atomic scattering factors from *SHELXL*93 (Sheldrick, 1993)
All H-atom parameters refined	

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

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

	x	y	z	U_{eq}
O1	0.8168 (2)	0.4839	0.2696 (2)	0.038 (1)
O2	0.8653 (4)	−0.0586 (8)	0.0535 (2)	0.064 (1)
O3	1.0886 (3)	0.1174 (8)	0.1948 (2)	0.063 (1)
O4	0.7920 (3)	1.1446 (8)	0.5261 (2)	0.045 (1)
N1	0.9158 (4)	0.1098 (9)	0.1390 (2)	0.045 (1)
N2	0.8180 (3)	0.9223 (8)	0.4431 (2)	0.037 (1)
C1	0.6377 (4)	0.6235 (10)	0.2922 (2)	0.038 (1)
C2	0.4776 (4)	0.5279 (9)	0.2132 (3)	0.046 (1)
C3	0.5575 (4)	0.3163 (10)	0.1381 (3)	0.046 (1)
C4	0.7612 (4)	0.2999 (9)	0.1757 (2)	0.039 (1)
C5	0.6468 (4)	0.8476 (9)	0.3854 (3)	0.039 (1)

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

O1—C4	1.347 (4)	N2—C5	1.272 (3)
O1—C1	1.366 (4)	C1—C2	1.358 (4)
O2—N1	1.230 (4)	C1—C5	1.441 (5)
O3—N1	1.224 (3)	C2—C3	1.397 (5)
O4—N2	1.382 (3)	C3—C4	1.348 (4)
N1—C4	1.413 (4)		
C4—O1—C1	104.8 (2)	O1—C1—C5	118.6 (2)
O3—N1—O2	124.0 (3)	C1—C2—C3	106.9 (3)
O3—N1—C4	118.9 (3)	C4—C3—C2	105.4 (3)
O2—N1—C4	117.0 (2)	O1—C4—C3	112.5 (3)
C5—N2—O4	111.8 (2)	O1—C4—N1	116.9 (2)
C2—C1—O1	110.4 (3)	C3—C4—N1	130.4 (3)
C2—C1—C5	130.9 (3)	N2—C5—C1	121.3 (3)

Computations were performed using the *SHELXL*93 package (Sheldrick, 1993).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1123). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-Nitrofuran-2-aldoxime

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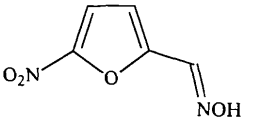
Abstract

The molecules of the title compound, $C_5H_4N_2O_4$, are linked through hydrogen bonds. Chains along the [100] direction are observed.

Comment

The present compound is one of a large series of heterocyclic nitro compounds that are being analysed in an attempt to elucidate the underlying structural parameters required for bioactivity. Although the molecular structure and crystal packing of the compound have been discussed (Matsuoka *et al.*, 1991), no atomic coordinates were reported. As the coordinates are necessary for performing molecular fitting between analogues, the crystal structure has been re-analysed.



A perspective view showing the atomic numbering scheme and hydrogen bonds is given in Fig. 1. The crystal contains well ordered molecules of 5-nitro-2-aldoxime in the form observed in crystals of 5-nitro-2-furaldehyde semicarbazone (Olszak, Peeters, Blaton & De Ranter, 1994). The bond lengths are similar to within 0.025 Å. The title compound has a *cis* conformation while the one cited is *trans* with respect to the double bond of the side chain (C21=N22). A comparison with the bond lengths given by Matsuoka *et al.* (1991) for the title compound shows they are the same to within 0.019 Å for bonds involving non-H atoms.

The planar C3=C2—C21=N22 group of atoms does not show conjugation between the double bonds. A comparison of the bond lengths with those given by Allen *et al.* (1987) and Burke-Laing & Laing (1976)