Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381 USA.

Molecular Structure Corporation (1988). *MSC/AFC Diffractometer Control Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.

Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Acta Cryst. (1994). **C50**, 1930–1931

trans-5-Nitro-2-furanacrylic Acid

TOMASZ A. OLSZAK, OSWALD M. PEETERS, NORBERT M. BLATON AND CAMIEL J. DE RANTER

Laboratorium voor Analytische Chemie en Medicinale Fysicochemie, Faculteit Farmaceutische Wetenschappen, Katholieke Universiteit Leuven, Van Evenstraat 4, B-3000 Leuven, Belgium

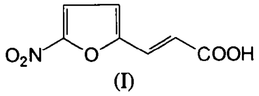
(*Received* 8 *December* 1993; *accepted* 4 *January* 1994)

Abstract

The molecules of the title compound, $C_7H_5NO_5$, are linked through hydrogen bonds to form a dimeric structure.

Comment

A perspective view showing the atomic numbering scheme and hydrogen bonds is given in Fig. 1. The crystal is composed of well ordered molecules of *trans*-5-nitro-2-furanacrylic acid, (I), in a form similar to the one observed in crystals of 2-(2′-dimethylaminovinyl)-5-nitrofuran (Kusa, Polynova, Porai-Koshits, Kovach & Vegkh, 1979). Both compounds are *trans* isomers with respect to the C21═C22 double bond. The molecule of the title compound is almost planar with no deviation from planarity greater than 0.05 Å. An examination of the torsion angles shows no deviation greater than 5° from either 0 or 180°.



(I)

The planar C3═C2—C21═C22 group shows no significant conjugation between the double bonds. The bond orders are 1.6, 1.2 and 2.0 for C3═C2, C2—C21 and C21═C22, respectively (Burke-Laing & Laing, 1976).

The molecules, related by an inversion, are linked into dimers by hydrogen bonds [O231···O232^{i} 1.76 (3) Å, O231—H231···O232^{i} 177 (2)°; symmetry code: (i) $1-x, -y, 2-z$]. The atoms involved in the hydrogen bonds form a planar eight-membered ring with deviations from planarity not greater than 0.02 Å. The ring makes a dihedral angle of 0.52 (3)° with the plane of the whole molecule.

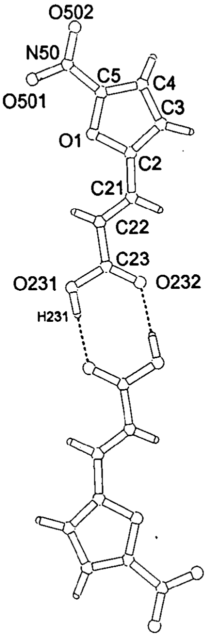


Fig. 1. The atomic numbering scheme and hydrogen bonds.

Experimental

The title compound was obtained from American Cyanamid Company (Pearl River, New York). Crystals were grown from an ethanol–acetonitrile (3:1) solution.

Crystal data

$C_7H_5NO_5$
$M_r = 183.12$
Monoclinic
$P2_1/n$
$a = 5.154$ (4) Å
$b = 14.253$ (5) Å
$c = 10.400$ (5) Å
$\beta = 99.61$ (6)°
$V = 753.3$ (8) Å^3
$Z = 4$
$D_x = 1.615$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 31 reflections
$\theta = 10.15$–$13.56°$
$\mu = 0.1320$ mm^{-1}
$T = 293$ K
Block
0.20 × 0.20 × 0.10 mm
Light brown

Data collection

Stoe Stadi-4 four-circle diffractometer
ω scans
Absorption correction: none
3631 measured reflections
1738 independent reflections
1075 observed reflections [$I > 2.0\sigma(I)$]

$R_{\rm int} = 0.03$
$\theta_{\rm max} = 27.5°$
$h = -7 \rightarrow 7$
$k = -19 \rightarrow 19$
$l = 0 \rightarrow 14$
4 standard reflections
frequency: 60 min
intensity variation: <3.0%

Refinement

Refinement on *F*
$R = 0.041$
$wR = 0.053$
$S = 1.033$
1075 reflections
138 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.0020F^2]$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.20$ e Å^{-3}
$\Delta\rho_{\min} = -0.36$ e Å^{-3}
Atomic scattering factors from *CRYSRULER* (Rizzoli, Sangermano, Calestani & Andreetti, 1989)

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

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

	x	y	z	$U_{\rm eq}$
O1	0.1267 (3)	0.4131 (1)	0.8735 (1)	0.0371 (4)
C2	−0.0253 (4)	0.3361 (1)	0.8335 (2)	0.0354 (6)
C21	0.0798 (4)	0.2447 (1)	0.8683 (2)	0.0369 (6)
C22	0.3126 (4)	0.2266 (1)	0.9404 (2)	0.0378 (6)
C23	0.3948 (4)	0.1286 (1)	0.9641 (2)	0.0348 (6)
O231	0.6363 (3)	0.1196 (1)	1.0300 (2)	0.0483 (5)
O232	0.2561 (3)	0.0613 (1)	0.9265 (1)	0.0449 (5)
C3	−0.2612 (4)	0.3638 (1)	0.7643 (2)	0.0410 (7)
C4	−0.2601 (4)	0.4626 (1)	0.7606 (2)	0.0424 (7)
C5	−0.0234 (4)	0.4876 (1)	0.8269 (2)	0.0357 (6)
N50	0.0889 (3)	0.5777 (1)	0.8549 (2)	0.0414 (5)
O501	0.3145 (4)	0.5830 (1)	0.9144 (2)	0.0640 (7)
O502	−0.0488 (3)	0.6453 (1)	0.8155 (2)	0.0584 (7)

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

O1—C2	1.372 (2)	C23—O232	1.221 (2)
O1—C5	1.355 (2)	C3—C4	1.409 (2)
C2—C21	1.434 (2)	C4—C5	1.345 (2)
C2—C3	1.364 (2)	C5--N50	1.419 (2)
C21—C22	1.330 (2)	N50—O501	1.225 (2)
C22—C23	1.469 (2)	N50—O502	1.226 (2)
C23—O231	1.323 (2)	O231—H231	0.90 (2)
C2—O1—C5	104.8 (2)	C2—C3—C4	107.3 (2)
O1—C2—C3	110.0 (2)	C3—C4—C5	105.0 (2)
O1—C2—C21	118.5 (2)	O1—C5—C4	113.0 (2)
C21—C2—C3	131.5 (2)	C4—C5—N50	130.5 (2)
C2—C21—C22	125.9 (2)	O1—C5—N50	116.6 (2)
C21—C22—C23	119.2 (2)	C5—N50—O502	116.7 (2)
C22—C23—O232	123.8 (2)	C5—N50—O501	118.7 (2)
C22—C23—O231	113.5 (2)	O501—N50—O502	124.6 (2)
O231—C23—O232	122.7 (2)	C23—O231—H231	110 (2)

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *PLUTON*93 (Spek, 1993). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

The authors thank American Cyanamid Company (Pearl River, New York) for providing the crystals. One of them (TO) is indebted to the Research Council of the K. U. Leuven (Belgium) for Senior Fellowship No. F/92/32.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, bond distances and angles involving H atoms and torsion angles have been deposited with the IUCr (Reference: NA1083). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Burke-Laing, M. & Laing, M. (1976). *Acta Cryst.* B**32**, 3216–3224
Kusa, A., Polynova, T. N., Porai-Koshits, M. A., Kovach, Ya. & Vegkh, D. (1979). *Zh. Strukt. Khim.* **20**, 556
Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
Rizzoli, C., Sangermano, V., Calestani, G. & Andreetti, G. D. (1989). *CRYSRULER*. Version 2.0. Univ. degli Studi di Parma, Italy.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination*. Univ. of Cambridge, England.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.
Spek, A. L. (1993). *PLUTON93. Molecular Graphics Program*. Univ. of Utrecht, The Netherlands.
Stoe & Cie (1992*a*). *DIF4. Diffractometer Control Program*. Version 7.0. Stoe & Cie, Darmstadt, Germany.
Stoe & Cie (1992*b*). *REDU4. Data Reduction Program*. Version 7.03. Stoe & Cie, Darmstadt, Germany.

Acta Cryst. (1994). C**50**, 1931–1933

2,6-Bis(dimethylamino)-3,5-pyridinedicarbaldehyde

Long-Li Lai, Ling-Kang Liu, Ming-Jen Shiao and Yuh-Sheng Wen

Institute of Chemistry, Academia Sinica, Nankang, Taipei, Taiwan

Abstract

The title molecule, $C_{11}H_{15}N_3O_2$, sits on a crystallographic twofold axis. This highly substituted pyridine compound has a very distorted pyridine plane (±0.1 Å) with the aldehyde O atoms displaced from the plane by *ca* 1.0 Å and the dimethylamino groups (planar within 0.06 Å) tilted away from the pyridine plane by 20.5 (1)°. The C1—C2 bond length [1.438 (3) Å] is much longer than that normally found in pyridine [1.379 Å; Allen, Kennard, Watson, Brammer, Orpen & Taylor (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19], as reasonably expected from consideration of steric effects.

Comment

The structure determination of the title compound (I) was carried out in order to obtain more structural parameters for 2,6-diaminopyridine derivatives and to see how