Acta Cryst. (1996). C**52**, 188–189

5-Nitro-2-furancarboxylic Acid

NATHANIEL W. ALCOCK,[a] WILLIAM ERRINGTON,[a] TERENCE J. KEMP[a] AND JANUSZ LECIEJEWICZ[b]

[a]*University of Warwick, Department of Chemistry, Coventry CV4 7AL, England, and* [b]*Institute of Nuclear Chemistry and Technology, 03-195 Warszawa, ul. Dorodna 16, Poland*

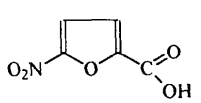
(*Received 24 May 1995; accepted 13 September 1995*)

Abstract

Almost planar molecules of the title compound, $C_5H_3NO_5$, are held together by hydrogen bonds of 2.604 (2) Å operating between the carboxylate O atoms of adjacent molecules giving rise to centrosymmetric dimers. The interactions between the dimers are much weaker, as indicated by the distances between the nearest atoms of adjacent moieties amounting to *ca* 3 Å.

Comment

Furoic acid (2-furancarboxylic acid) is known to form metal complexes which exhibit a variety of coordination schemes (Paluchowska, Lis & Leciejewicz, 1994, and references therein). The influence of large substituents attached to the furan ring upon the coordination characteristics of this ligand is also of interest; hence, the structure of the title compound, (I), is now reported as a prelude to a study of its coordination chemistry.



(I)

The molecular structure of the title compound is illustrated in Fig. 1. Dimers are formed by hydrogen bonding between adjacent carboxylate O atoms with an O—H···O distance of 2.604 (2) Å and a bond angle of 174 (2)° at H. The dimers are probably held together by weak van der Waals interactions as indicated by the closest intermolecular distances of *ca* 3 Å. The O atoms of the nitro groups do not participate in the hydrogen bonding. The furan ring is planar within experimental error; the nitro and carboxylic moieties make dihedral angles of *ca* 4° with the furan plane. The r.m.s. deviation from the least-squares plane through all non-H atoms is 0.044 Å.

Similar hydrogen-bonding patterns have been observed in the crystal structures of 2-furancarboxylic acid (Gilmore, Mallinson & Speakman, 1983) and 3-furancarboxylic acid (Paluchowska, Maurin & Leciejewicz, 1995). The bond lengths and angles observed in the title compound compare well with those reported for the above acids.

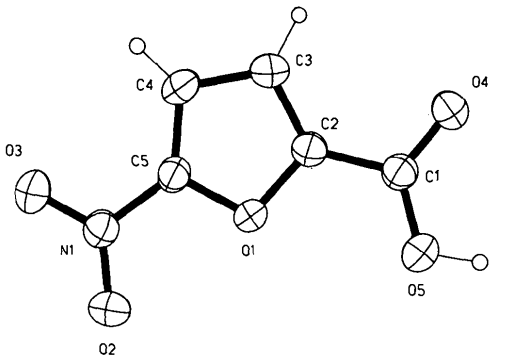


Fig. 1. View of the molecule showing the atomic numbering. Displacement ellipsoids are drawn at the 50% probability level.

Experimental

A commercial sample of the title compound was recrystallized from an aqueous solution.

Crystal data

$C_5H_3NO_5$
M_r = 157.08
Monoclinic
$C2/c$
a = 22.162 (17) Å
b = 5.565 (4) Å
c = 10.570 (11) Å
β = 109.33 (7)°
V = 1230.1 (18) Å^3
Z = 8
D_x = 1.696 Mg m^{-3}
D_m = 1.72 (2) Mg m^{-3}
D_m measured by flotation in bromobenzene/bromoform
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 27 reflections
θ = 9–14°
μ = 0.157 mm^{-1}
T = 220 (2) K
Plate
0.46 × 0.37 × 0.10 mm
Colourless

Data collection

Siemens *P3R3* diffractometer
ω–2θ scans
Absorption correction: none
1370 measured reflections
1098 independent reflections
943 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0187
θ_{max} = 25.04°
h = 0 → 26
k = −1 → 6
l = −12 → 11
3 standard reflections monitored every 200 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0355
$wR(F^2)$ = 0.0958
S = 1.050
1098 reflections
110 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2 + 0.9226P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.175 e Å^{-3}
$\Delta\rho_{min}$ = −0.225 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.031 (3)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

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

	x	y	z	U_{eq}
O1	0.37829 (5)	0.0838 (2)	0.85350 (11)	0.0313 (3)
O2	0.32206 (7)	0.4750 (2)	0.88946 (13)	0.0447 (4)
O3	0.27026 (7)	0.5054 (2)	0.67691 (14)	0.0448 (4)
O4	0.46017 (7)	−0.4518 (3)	0.83946 (13)	0.0461 (4)
O5	0.46085 (6)	−0.2286 (3)	1.01629 (12)	0.0439 (4)
N1	0.30843 (7)	0.4061 (3)	0.77363 (14)	0.0338 (4)
C1	0.44395 (8)	−0.2734 (3)	0.8913 (2)	0.0331 (4)
C2	0.40099 (8)	−0.1051 (3)	0.8002 (2)	0.0321 (4)
C3	0.37659 (9)	−0.1025 (4)	0.6649 (2)	0.0382 (5)
C4	0.33612 (9)	0.0977 (4)	0.6292 (2)	0.0370 (5)
C5	0.33927 (8)	0.2000 (3)	0.7461 (2)	0.0307 (4)

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

O1—C5	1.343 (2)	N1—C5	1.414 (2)
O1—C2	1.365 (2)	C1—C2	1.451 (3)
O2—N1	1.222 (2)	C2—C3	1.351 (3)
O3—N1	1.222 (2)	C3—C4	1.401 (3)
O4—C1	1.244 (2)	C4—C5	1.341 (3)
O5—C1	1.272 (3)		
C5—O1—C2	104.12 (14)	C3—C2—C1	130.9 (2)
O2—N1—O3	124.7 (2)	O1—C2—C1	118.2 (2)
O2—N1—C5	119.00 (15)	C2—C3—C4	106.9 (2)
O3—N1—C5	116.3 (2)	C5—C4—C3	104.8 (2)
O4—C1—O5	125.8 (2)	C4—C5—O1	113.4 (2)
O4—C1—C2	116.4 (2)	C4—C5—N1	130.7 (2)
O5—C1—C2	117.8 (2)	O1—C5—N1	115.88 (15)
C3—C2—O1	110.8 (2)		
O5—C1—C2—C3	178.2 (2)	O3—N1—C5—O1	−176.23 (14)
O4—C1—C2—O1	175.27 (15)		

The temperature of the crystal was controlled using the Oxford Cryosystems Cryostream Cooler (Cosier & Glazer, 1986). H atoms were added from difference density maps. Anisotropic displacement parameters were used for all non-H atoms; H atoms were given isotropic displacement parameters equal to 1.2 times the equivalent isotropic displacement parameter of the atom to which they are attached.

Data collection: Siemens *P3R3* system. Cell refinement: Siemens *P3R3* system. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993).

We wish to acknowledge the use of the Cambridge Structural Database (Allen *et al.*, 1991) through the EPSRC's Chemical Database Service at Daresbury. One of us (JL) wishes to thank the Department of Chemistry at the University of Warwick for its hospitality.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, torsion angles and complete geometry have been deposited with the IUCr (Reference: CF1029). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Davies, J. E., Galloy, J. J., Johnson, O., Kennard, O., Macrae, C. F., Mitchell, E. M., Mitchell, G. F., Smith, J. M. & Watson, D. G. (1991). *J. Chem. Inf. Comput. Sci.* **31**, 187–204.

Cosier, J. & Glazer, A. M. (1986). *J. Appl. Cryst.* **19**, 105–107.

Gilmore, C. J., Mallinson, P. R. & Speakman, J. C. (1983). *Acta Cryst.* C**39**, 1111–1113.

Paluchowska, B., Lis, T. & Leciejewicz, J. (1994). *Acta Cryst.* C**50**, 683–686.

Paluchowska, B., Maurin, J. & Leciejewicz, J. (1995). *Acta Cryst.* C**51**. In the press.

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1993). *SHELXL93. Program for Crystal Structure Refinement*. University of Göttingen, Germany.

Acta Cryst. (1996). C**52**, 189–191

1-Trityl-4-nitroimidazole

EWA SKRZYPCZAK-JANKUN[a] AND RAVI G. KURUMBAIL[b]†

[a]*Department of Chemistry, University of Toledo, Toledo, OH 43606, USA, and* [b]*Howard Hughes Medical Institute, University of Texas, Dallas, TX 75235, USA*

(*Received 31 October 1994; accepted 24 July 1995*)

Abstract

X-ray analysis confirmed the configuration of the title N1-alkylated C4-nitroimidazole inhibitor. The plane of the imidazole ring, sitting on an axis of the trityl propeller, bisects the angle between two phenyl rings, while the nitro group extends over the third. Modeling of the interactions between the cytochrome P450 and the title compound ($C_{22}H_{17}N_3O_2$) has been performed on the basis of the crystal structures of 1-trityl-4-nitroimidazole and bacterial cytochrome $P450_{BM-3}$. The replacements and deletions in the sequence of the latter has been performed to match mammalian cytochrome P450-IIIA1. The modeling explained why inhibitors with a C4-substituted imidazole ring showed lower effectivity than those without substituents, as an additional group of atoms at C4 prevents close interactions of the imidazole ring with the heme Fe atom.

Comment

Tritylimidazoles are used clinically as topical antifungal agents (von Buchel, Draber, Regel & Pempel, 1972). The antifungal activity is thought to be due to inhibition of a fungal cytochrome P450 mixed-function oxidase, which catalyses 14-α-dimethylation of sterols in the conversion of lanosterol to ergosterol. Tritylimidazoles also selectively inhibit certain mammalian cytochrome P450 isozymes (Rodrigues, Gibson, Ioannides & Parke, 1987). The structures of substituted tritylimidazoles such

† Present address: Monsanto/Searle BB4K, 700 Chesterfield Village Parkway North, St Louis, MO 63198, USA.