C2	−0.328 (1)	0.3392 (7)	−0.2883 (9)	7.2 (2)
C3	−0.090 (1)	0.3561 (7)	−0.2888 (8)	5.6 (2)
C4	0.036 (1)	0.2648 (6)	−0.2883 (8)	5.2 (2)
C5	0.072 (1)	0.2035 (6)	−0.1733 (8)	5.4 (2)
C6	0.183 (1)	0.1180 (7)	−0.182 (1)	7.1 (3)
C7	0.248 (1)	0.0938 (7)	−0.301 (1)	7.3 (3)
C8	0.211 (2)	0.1551 (8)	−0.413 (1)	7.8 (3)
C9	0.107 (1)	0.2410 (8)	−0.4085 (8)	5.9 (2)
C10	−0.478 (1)	0.3843 (7)	−0.4160 (8)	5.7 (2)
C11	−0.484 (1)	0.3511 (7)	−0.5536 (9)	6.3 (2)
C12	−0.596 (1)	0.3934 (9)	−0.6645 (9)	7.5 (3)
C13	−0.717 (1)	0.4735 (9)	−0.639 (1)	8.4 (3)
C14	−0.707 (1)	0.5110 (8)	−0.509 (1)	7.4 (3)
C15	−0.587 (1)	0.4663 (7)	−0.3946 (9)	6.8 (3)
C16	0.068 (1)	0.4457 (8)	−0.0700 (9)	7.0 (3)
C17	0.102 (1)	0.5496 (7)	−0.1035 (8)	6.0 (2)
C18	−0.042 (1)	0.6063 (7)	−0.1834 (9)	6.3 (2)
C19	−0.003 (2)	0.7049 (8)	−0.2100 (9)	7.8 (3)
C20	0.187 (2)	0.7431 (8)	−0.157 (1)	8.1 (3)
C21	0.331 (2)	0.6878 (9)	−0.081 (1)	9.7 (4)
C22	0.289 (1)	0.5926 (9)	−0.054 (1)	8.6 (3)

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

O1—C1	1.26 (1)	C10—C11	1.41 (1)
N1—C1	1.33 (1)	C10—C15	1.38 (1)
N1—C3	1.41 (1)	C11—C12	1.34 (2)
N1—C16	1.38 (1)	C12—C13	1.42 (2)
C1—C2	1.50 (2)	C13—C14	1.36 (2)
C2—C3	1.64 (2)	C14—C15	1.40 (2)
C2—C10	1.59 (1)	C16—C17	1.48 (2)
C3—C4	1.51 (1)	C17—C18	1.38 (2)
C4—C5	1.39 (1)	C17—C22	1.41 (2)
C4—C9	1.38 (1)	C18—C19	1.41 (2)
C5—C6	1.40 (1)	C19—C20	1.41 (2)
C6—C7	1.35 (2)	C20—C21	1.35 (2)
C7—C8	1.36 (2)	C21—C22	1.37 (2)
C8—C9	1.38 (2)		
C1—N1—C3	98 (1)	C4—C9—C8	119 (1)
C1—N1—C16	132 (1)	C2—C10—C11	122 (1)
C3—N1—C16	130 (1)	C2—C10—C15	119 (1)
O1—C1—N1	130 (1)	C11—C10—C15	118 (1)
O1—C1—C2	136 (1)	C10—C11—C12	123 (1)
N1—C1—C2	94 (1)	C11—C12—C13	117 (1)
C1—C2—C3	82.3 (9)	C12—C13—C14	121 (1)
C1—C2—C10	122.1 (9)	C13—C14—C15	120 (1)
C3—C2—C10	116.6 (8)	C10—C15—C14	119 (1)
N1—C3—C2	85.7 (8)	N1—C16—C17	115 (1)
N1—C3—C4	115 (1)	C16—C17—C18	122 (1)
C2—C3—C4	116.2 (9)	C16—C17—C22	119 (1)
C3—C4—C5	121.7 (9)	C18—C17—C22	118 (1)
C3—C4—C9	118 (1)	C17—C18—C19	120 (1)
C5—C4—C9	120 (1)	C18—C19—C20	119 (1)
C4—C5—C6	119 (1)	C19—C20—C21	122 (1)
C5—C6—C7	121 (1)	C20—C21—C22	119 (2)
C6—C7—C8	120 (1)	C17—C22—C21	122 (1)
C7—C8—C9	122 (1)		

H atoms were included in the refinement at calculated positions with C—H = 0.95 Å.

Data collection and cell refinement: *CAD*-4 *Software* (Enraf–Nonius, 1989). The structure was solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). Full-matrix least-squares refinement was carried out using *MolEN* (Fair, 1990). Program used to prepare figure: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: PA1165). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Alcaide, B., Leon-Santiago, M. A., Perez-Ossioro, R., Plumet, J., Assierra, M. & de la Torre, M. C. (1982). *Synthesis*, pp. 989–990.

Boyd, D. B. (1983). *J. Med. Chem.* **26**, 1010.

Cohen, N. C. (1983). *Actual. Chim.* pp. 7–10.

Enraf–Nonius (1989). *CAD-4 Software*. Version 5. Enraf–Nonius, Delft, The Netherlands.

Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis.* Enraf–Nonius, Delft, The Netherlands.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Universities of York, England, and Louvain, Belgium.

Pain, S., Biswas, G., Bose, A. & Banerjee, A. (1992). *Acta Cryst.* C**48**, 666–669.

Takasuka, M., Nishikawa, J. & Tori, K. (1982). *J. Antibiot.* **35**, 1729–1733.

Vinković, M. & Herak, J. J. (1993). *Acta Cryst.* C**49**, 1663–1665.

Acta Cryst. (1996). C**52**, 203–205

2-Cyano-*N*-furfuryl-3-(2-furyl)acrylamide

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(*Received 28 September 1993; accepted 26 June 1995*)

Abstract

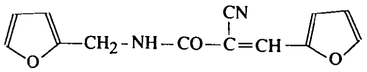
In the title compound, $C_{13}H_{10}N_2O_3$, the two equivalent molecules in the asymmetric unit form dimers by means of an intermolecular N—H···N hydrogen bond with an N···H distance of 2.42 (2) Å. There are weak intramolecular and intermolecular interactions between the C—H···N and C—H···O atoms. There are no unusual intramolecular distances or angles. The furan rings are rotated 81.1 (2)° relative to each other.

Comment

It is not surprising that many natural products exhibit some bioactivity. In most cases, however, they are not sufficiently active or are too toxic to be of immediate practical use. In such cases, the structures have to be modified for activity enhancement or toxicity reduction, either empirically or more logically, *e.g.* by quantitative structure–activity relationship (QSAR) studies (Clementi, 1984; Franke, 1984).

In recent years furan derivatives obtained by synthetic procedures or as constituents of naturally occurring molecules have been studied and used as drugs showing antimicrobiotic hypotensive and antitumor activity (Bartroli, Lami, Quincoses & Peseke, 1984). The chemical and biological characteristics of these substances can be directly related to the conformations of their molecules (Bartroli, 1985).

The present structure determination was undertaken to provide information concerning the preferred conformation of the title molecule, (I). This information will be used in multivariate QSAR studies of a series of furan derivatives studied using crystallographic and molecular-modelling methods (Pomés Hernández, Duque Rodríguez, Villena Rodríguez & Soriano Garcia, 1995).



(I)

There are no unusual intramolecular distances or angles. The two equivalent molecules in the asymmetric unit form dimers by means of an N—H···N hydrogen bond with an H(1)···N(2)(2 − x, −y, 2 − z) distance of 2.42 (2) Å. Table 3 shows the intramolecular and intermolecular weak interactions between the C—H···N and C—H···O atoms. The furan ring O(1), C(2), C(3), C(4), C(5) is rotated 81.1 (2)° with respect to the furan ring C(10), C(11), C(12), C(13), O(5).

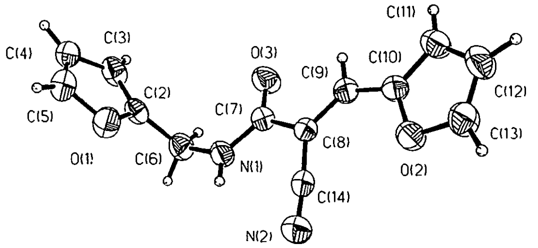


Fig. 1. View of a molecule of the title compound with the atomic numbering (*SHELXTL-Plus*; Sheldrick, 1987). Displacement ellipsoids are drawn at the 50% probability level.

Experimental

The title compound was obtained in 75% yield from the reaction of *N*-fur-2-ylmethylcyanacetamide and furfuraldehyde (a derivative of sugar cane by-products) (Bartroli & Lami, 1995). Recrystallization by slow evaporation from ethanol gave light-yellow needles (m.p. 407–408 K).

Crystal data

$C_{13}H_{10}N_2O_3$
$M_r = 242.23$
Triclinic
$P\bar{1}$
$a = 5.311\ (1)$ Å
$b = 9.495\ (1)$ Å
$c = 11.442\ (2)$ Å
$\alpha = 94.13\ (2)°$
$\beta = 93.79\ (2)°$
$\gamma = 93.60\ (2)°$
$V = 572.9\ (3)$ Å^3
$Z = 2$
$D_x = 1.40$ Mg m^{-3}
$D_m = 1.39$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 37 reflections
$\theta = 11.5–19°$
$\mu = 0.10$ mm^{-1}
$T = 298$ K
Needle
0.6 × 0.4 × 0.1 mm
Light yellow

Data collection

Siemens *P*3/*PC* diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2268 measured reflections
2028 independent reflections
1292 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int} = 0.04$
$\theta_{\rm max} = 25°$
$h = 0 \rightarrow 6$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 13$
3 standard reflections monitored every 100 reflections intensity decay: <2%

Refinement

Refinement on F^2
$R(F) = 0.041$
$wR(F^2) = 0.039$
$S = 1.17$
1292 reflections
195 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.000413F^2]$

$(\Delta/\sigma)_{\rm max} = 0.54$
$\Delta\rho_{\rm max} = 0.17$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}
Extinction correction: none
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)*

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

	x	y	z	$U_{\rm eq}$
O(1)	0.7583 (3)	0.4082 (2)	0.9034 (2)	0.053 (1)
O(2)	1.1503 (4)	−0.2717 (2)	0.6210 (2)	0.051 (1)
O(3)	0.4448 (4)	0.0676 (2)	0.6728 (2)	0.054 (1)
N(1)	0.6267 (4)	0.1025 (2)	0.8564 (2)	0.044 (1)
N(2)	1.0973 (5)	−0.1199 (2)	0.8928 (2)	0.056 (1)
C(2)	0.5213 (5)	0.3524 (3)	0.8661 (2)	0.039 (1)
C(3)	0.3935 (6)	0.4511 (3)	0.8163 (2)	0.049 (1)
C(4)	0.5545 (6)	0.5763 (3)	0.8226 (2)	0.052 (1)
C(5)	0.7701 (6)	0.5463 (3)	0.8749 (3)	0.054 (1)
C(6)	0.4526 (6)	0.2055 (3)	0.8949 (3)	0.047 (1)
C(7)	0.6084 (5)	0.0412 (2)	0.7466 (2)	0.040 (1)
C(8)	0.8017 (5)	−0.0619 (2)	0.7180 (2)	0.037 (1)
C(9)	0.8099 (5)	−0.1139 (3)	0.6054 (2)	0.041 (1)
C(10)	0.9719 (5)	−0.2092 (3)	0.5535 (2)	0.040 (1)
C(11)	0.9904 (6)	−0.2519 (3)	0.4391 (2)	0.054 (1)
C(12)	1.1859 (6)	−0.3433 (3)	0.4338 (3)	0.058 (1)
C(13)	1.2765 (6)	−0.3522 (3)	0.5441 (3)	0.058 (1)
C(14)	0.9726 (5)	−0.0980 (2)	0.8120 (2)	0.040 (1)

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

O(1)—C(2)	1.364 (3)	O(1)—C(5)	1.372 (3)
O(2)—C(10)	1.374 (3)	O(2)—C(13)	1.361 (4)
O(3)—C(7)	1.221 (3)	N(1)—C(6)	1.453 (4)
N(1)—C(7)	1.342 (3)	N(2)—C(14)	1.140 (3)
C(2)—C(3)	1.331 (4)	C(2)—C(6)	1.485 (4)
C(3)—C(4)	1.414 (4)	C(4)—C(5)	1.314 (4)

C(7)—C(8)	1.498 (4)	C(8)—C(9)	1.350 (3)
C(8)—C(14)	1.435 (3)	C(9)—C(10)	1.415 (4)
C(10)—C(11)	1.353 (4)	C(11)—C(12)	1.396 (4)
C(12)—C(13)	1.331 (4)		
C(2)—O(1)—C(5)	106.3 (2)	C(10)—O(2)—C(13)	105.7 (2)
C(6)—N(1)—C(7)	121.9 (2)	O(1)—C(2)—C(3)	109.0 (2)
O(1)—C(2)—C(6)	117.1 (2)	C(3)—C(2)—C(6)	133.7 (2)
C(2)—C(3)—C(4)	107.7 (3)	C(3)—C(4)—C(5)	106.4 (3)
O(1)—C(5)—C(4)	110.5 (3)	N(1)—C(6)—C(2)	114.3 (2)
O(3)—C(7)—N(1)	122.4 (2)	O(3)—C(7)—C(8)	121.2 (2)
N(1)—C(7)—C(8)	116.4 (2)	C(7)—C(8)—C(9)	118.6 (2)
C(7)—C(8)—C(14)	118.0 (2)	C(9)—C(8)—C(14)	123.4 (2)
C(8)—C(9)—C(10)	130.8 (2)	O(2)—C(10)—C(9)	120.9 (2)
O(2)—C(10)—C(11)	109.0 (2)	C(9)—C(10)—C(11)	130.1 (3)
C(10)—C(11)—C(12)	107.6 (3)	C(11)—C(12)—C(13)	106.3 (3)
O(2)—C(13)—C(12)	111.4 (3)	N(2)—C(14)—C(8)	174.3 (3)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N(1)—H(1)···N(2)	0.82 (3)	2.80 (3)	3.396 (3)	131 (2)
	1.03*	2.67*		128*
C(6)—H(5)···O(3)	1.01 (3)	2.53 (3)	2.770 (4)	92 (1)
	1.08*	2.53*		91*
C(9)—H(7)···O(3)	0.94 (3)	2.41 (3)	2.784 (4)	103 (2)
	1.08*	2.39*		100*
N(1)—H(1)···N(2^{i})	0.82 (3)	2.42 (2)	3.123 (3)	145 (2)
C(4)—H(3)···O(2^{ii})	0.96 (3)	2.79 (3)	3.497 (3)	130 (2)
	1.08*	2.71*		129*
C(5)—H(4)···N(2^{iii})	0.94 (3)	2.78 (3)	3.504 (3)	134 (2)
	1.08*	2.69*		132*
C(9)—H(7)···O(3^{iv})	0.94 (3)	2.63 (2)	3.444 (3)	145 (2)
	1.08*	2.52*		144*
C(11)—H(8)···O(3^{iv})	0.89 (3)	2.45 (3)	3.238 (3)	146 (2)
	1.08*	2.30*		144*

Symmetry codes: (i) $2 - x, -y, 2 - z$; (ii) $x - 1, 1 + y, z$; (iii) $x, 1 + y, z$; (iv) $1 - x, -y, 1 - z$.

* Normalized values (Jeffrey & Lewis, 1978; Taylor & Kennard, 1983).

All computations were performed on an IBM AT486 computer.

Data collection: *XSCANS* (Siemens, 1992). Cell refinement: *SHELXTL-Plus* (Sheldrick, 1987). Data reduction: *SHELXTL-Plus*. Program(s) used to solve structure: *SHELXTL-Plus* (direct methods).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, torsion angles and complete geometry have been deposited with the IUCr (Reference: AB1126). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bartroli, R. (1985). PhD dissertation, Central University of VillaClara, Cuba.

Bartroli, R. & Lami, L. (1995). In preparation.

Bartroli, R., Lami, L., Quincoses, J. & Peseke, K. (1984). Certificado de autor de invención (Cuban Patent) No. 21789, La Habana, Cuba.

Clementi, S. (1984). *Drug Design: Fact or Fantasy?* edited by G. Jolles & K. R. H. Wooldridge, pp. 73–75. London: Academic Press.

Franke, R. (1984). *Theoretical Drugs Design Methods*, pp. 188–190. Amsterdam: Elsevier.

Jeffrey, G. A. & Lewis, L. (1978). *Carbohydr. Res.* **60**, 179–180.

Pomés Hernández, R., Duque Rodríguez, J., Villena Rodríguez, R. & Soriano Garcia, M. (1995). *Acta Cryst.* C**51**, 1575–1577.

Sheldrick, G. M. (1987). *SHELXTL-Plus*. PC Version. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1992). *XSCANS. X-ray Single Crystal Analysis System.* Version 2.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Taylor, R. & Kennard, O. (1983). *Acta Cryst.* B**39**, 133–134.

Acta Cryst. (1996). C**52**, 205–207

Pyo$_2$[18]diene-N$_6$.4HBr.H$_2$O

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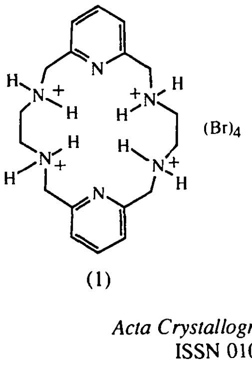
(*Received 27 September 1994; accepted 17 July 1995*)

Abstract

The X-ray crystal structure of 3,6,14,17,23,24-hexaazatricyclo[17.3.1.1^{8,12}]tetracosa-1(23),8,10,12(24),19,21-hexene tetrahydrobromide monohydrate, $C_{18}H_{30}N_6^{4+}$.$4Br^-$.H_2O, was determined. The compound is monomeric and the 18-membered macrocyclic ring contains four saturated amine and two pyridine units. The pyridine rings are inclined so that the ring N atoms are pointed above and below the centroid of the macrocycle.

Comment

The title compound (1) was prepared as described in the literature (Rothermel, Miao, Hill & Jackels, 1992) and its structure was determined in order to compare its features with those found for various metal complexes of the ligand (Bryant, Lachgar, Coates & Jackels, 1994). The compound crystallized in a monoclinic space group with four molecules per unit cell. Each molecule consists of a macrocyclic tetracation, pyo$_2$[18]-diene-N$_6$H$_4^{4+}$, four Br$^-$ anions, and a water molecule. The macrocyclic ring sits on a crystallographic center of symmetry (1/4,1/4,0), the water molecule resides on a crystallographic twofold axis and the two distinct Br$^-$ atoms are in general positions. The macrocyclic ring is puckered with N(1) (−0.043 Å), C(9) (−0.236 Å), N(2) (−0.612 Å) and C(6) (−0.510 Å) lying below the best plane through the macrocyclic backbone and C(1) (0.432 Å), N(3) (0.165 Å), C(8) (0.476 Å), C(7) (0.685 Å), and C(5) (0.110 Å) lying above that plane. The symmetry and puckering result in the N-atom lone pair on each pyridine ring being tipped out of the macrocycle cavity in directions opposite to each other.



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Acta Crystallographica Section C
ISSN 0108-2701 ©1996