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*Acta Cryst.* (1990). **C46**, 106–108

## Structure of (*R*)-Tetrahydro-2-furancarboxylic Acid Monohydrate

BY SACHIKO BANDO AND TSUNEHIRO TAKANO\*

*Setsunan University, Faculty of Pharmaceutical Sciences, 45-1 Nagaotoge-cho, Hirakata, Osaka 573-01, Japan*

AND RIE TANAKA, HIROMITSU IWATA AND MASAJI ISHIGURO

*Suntory Institute for Biomedical Research, 1-1 Wakayamadai, Shimamoto, Osaka 618, Japan*

(Received 27 January 1989; accepted 13 April 1989)

**Abstract.**  $C_5H_{10}O_4$ ,  $M_r = 134.31$ , monoclinic,  $P2_1$ ,  $a = 11.670$  (2),  $b = 6.177$  (3),  $c = 10.704$  (2) Å,  $\beta = 115.11$  (2)°,  $V = 698.7$  (4) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.26$ ,  $D_x = 1.28$  g cm<sup>-3</sup>, Cu  $K\alpha$ ,  $\lambda = 1.54178$  Å,  $\mu = 9.69$  cm<sup>-1</sup>,  $F(000) = 288$ , room temperature,  $R = 0.046$  for 1047 reflections with  $|F_o| > 3\sigma(F)$ . There are two independent molecules of the title compound in the crystal. No twofold symmetry is observed in the tetrahydrofuran rings. Hydrogen bonds between water molecules and carboxylic acid groups stabilize the crystal structure.

**Introduction.** In the course of development of penem antibiotics, we have found that compounds with a tetrahydrofuran ring attached to the penem nucleus show excellent activity against a wide range of bacteria (Ishiguro, Iwata, Nakatsuka, Tanaka, Maeda, Nishihara, Noguchi & Nishino, 1988). The structural importance of the tetrahydrofuran ring has also been found in polyether antibiotics (Agtarap, Chamberlin, Pinkerton & Steinrauf, 1967).

(*R*)-Tetrahydro-2-furancarboxylic acid (Belanger & Williams, 1983) is one of the smallest tetrahydrofuran derivatives, which have a characteristic amphiphilic structure. The accurate structure of the

molecule should be useful for understanding the interactions of the molecule with water molecules and biological macromolecules.

**Experimental.** The title compound was crystallized from tetrahydrofuran solution.  $D_m$  by flotation in chlorobenzene/bromobenzene. The crystals are deliquescent.

Diffraction intensities were measured using a colorless hexagonal-plate crystal,  $0.15 \times 0.3 \times 0.7$  mm, sealed in a quartz capillary tube;  $\omega$ - $2\theta$  scan mode with variable scan width; maximum  $2\theta = 120^\circ$ ; Rigaku-AFC diffractometer and Ni-filtered Cu  $K\alpha$  radiation. Lattice parameters and orientation matrix refined using 20 reflections in the range  $20 < \theta < 21^\circ$  by the least-squares method. 1138 unique reflections measured in the index range  $-13 \leq h \leq 13$ ,  $0 \leq k \leq 6$ ,  $0 \leq l \leq 12$ . 1047 reflections with  $|F_o| > 3\sigma(F)$ . Three standard reflections (004,  $3\bar{1}\bar{3}$ , 420) were monitored every 100 reflections. Mean variation of the standard 0.3%. Intensities corrected for absorption by the method of North, Phillips & Mathews (1968) using two reflections; normalized transmission factors 0.99–0.88. No extinction correction.

The structure was solved by direct phase determination using the *RANTAN* option of *MULTAN87* (Debaerdemaeker, Germain, Main,

\* To whom correspondence should be addressed.

Table 1. Final fractional coordinates and equivalent isotropic thermal parameters with *e.s.d.*'s in parentheses
$$B_{\text{eq}} = \frac{1}{3}[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab(\cos\gamma)\beta_{12} + ac(\cos\beta)\beta_{13} + bc(\cos\alpha)\beta_{23}]$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{eq}}(\text{\AA}^2)$
<b>Molecule A</b>				
O(1)	0.4751 (3)	0.1211 (0)	0.7807 (3)	4.6 (1)
C(2)	0.3686 (4)	0.0179 (11)	0.6767 (4)	3.6 (2)
C(3)	0.4133 (5)	-0.0755 (14)	0.5736 (6)	5.2 (2)
C(4)	0.5556 (5)	-0.0693 (14)	0.6496 (6)	5.2 (2)
C(5)	0.5768 (5)	0.1212 (14)	0.7406 (6)	5.5 (2)
C(6)	0.2553 (4)	0.1669 (11)	0.6110 (4)	3.4 (2)
O(7)	0.2674 (3)	0.3666 (9)	0.6330 (3)	4.5 (1)
O(8)	0.1542 (3)	0.0730 (9)	0.5345 (3)	4.5 (1)
<b>Molecule B</b>				
O(11)	0.0243 (3)	-0.1102 (10)	0.8594 (4)	5.7 (2)
C(12)	0.1113 (4)	0.0585 (11)	0.9298 (5)	3.9 (2)
C(13)	0.0614 (6)	0.2563 (14)	0.8383 (8)	6.1 (3)
C(14)	-0.0821 (8)	0.2149 (20)	0.7720 (10)	9.1 (5)
C(15)	-0.0957 (6)	-0.0236 (19)	0.7729 (10)	8.5 (4)
C(16)	0.2443 (4)	-0.0068 (11)	0.9526 (4)	3.5 (2)
O(17)	0.2598 (3)	-0.1787 (9)	0.9019 (3)	4.6 (1)
O(18)	0.3304 (3)	0.1233 (9)	1.0221 (4)	5.0 (1)
<b>Water molecules</b>				
O(19)	0.4191 (3)	0.4871 (9)	0.9151 (4)	5.2 (2)
O(20)	0.0944 (4)	-0.3106 (10)	0.6297 (4)	5.5 (2)

Tate & Woolfson, 1987). The phase set, of RESID = 15.39, gave all non-H atoms except the water O atoms. Refinement by blocked-diagonal-matrix least squares with  $|F_o| > 3\sigma(F)$  to  $(\sin\theta/\lambda)_{\text{max}} = 0.562 \text{ \AA}^{-1}$  revealed two water molecules. All non-H atoms with anisotropic thermal parameters and H atoms found on difference maps with isotropic thermal parameters were refined by full-matrix least squares with  $w = 1.0$  for  $|F_o| \leq 8.0$ ,  $w = (8.0/|F_o|)^2$  for  $|F_o| > 8.0$ . Final  $R = 0.046$ ,  $wR = 0.059$ ,  $S = 0.14$ , max.  $\Delta/\sigma = 0.17$  for isotropic B of H(141), and max. and min. electron density in the final difference map 0.19 and  $-0.18 \text{ e \AA}^{-3}$ . Atomic scattering factors from *International Tables for X-ray Crystallography* (1962). Calculations were carried out on NEC PC-9801 personal computers using local unpublished programs.

**Discussion.** The atomic parameters are given in Table 1.\* Bond lengths, bond angles and torsion angles are presented in Table 2, and the distances between O atoms within hydrogen-bond lengths in Table 3. The structures of the two independent molecules with the atomic numbering scheme are shown in Fig. 1. A stereoscopic view of the packing is given in Fig. 2.

\* Lists of structure factors, anisotropic temperature factors, H-atom parameters and geometrical data of H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52147 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths ( $\text{\AA}$ ), bond angles ( $^\circ$ ) and torsion angles ( $^\circ$ ) with *e.s.d.*'s in parentheses

<b>Molecule A</b>			
O(1)—C(2)	1.420 (8)	O(1)—C(5)	1.424 (9)
C(2)—C(3)	1.520 (11)	C(2)—C(6)	1.517 (9)
C(3)—C(4)	1.508 (12)	C(4)—C(5)	1.480 (12)
C(6)—O(7)	1.252 (8)	C(6)—O(8)	1.257 (8)
C(2)—O(1)—C(5)	109.1 (5)	O(1)—C(2)—C(3)	106.5 (6)
O(1)—C(2)—C(6)	113.0 (5)	C(3)—C(2)—C(6)	113.2 (6)
C(2)—C(3)—C(4)	103.8 (7)	C(3)—C(4)—C(5)	102.3 (7)
O(1)—C(5)—C(4)	105.7 (7)	C(2)—C(6)—O(7)	120.3 (6)
C(2)—C(6)—O(8)	114.5 (6)	O(7)—C(6)—O(8)	125.2 (6)
O(7)—C(6)—C(2)—O(1)	11.7 (2)	O(8)—C(6)—C(2)—O(1)	-169.4 (7)
C(6)—C(2)—O(1)—C(5)	-117.9 (6)	C(2)—O(1)—C(5)—C(4)	-27.3 (3)
O(1)—C(5)—C(4)—C(3)	35.8 (4)	C(5)—C(4)—C(3)—C(2)	-30.6 (4)
C(4)—C(3)—C(2)—C(6)	140.1 (7)	C(3)—C(2)—C(6)—C(7)	-109.6 (8)
C(3)—C(2)—C(6)—O(8)	69.4 (7)	C(3)—C(2)—O(1)—C(5)	7.1 (6)
C(4)—C(3)—C(2)—O(1)	15.2 (4)		
<b>Molecule B</b>			
O(11)—C(12)	1.427 (9)	O(11)—C(15)	1.416 (13)
C(12)—C(13)	1.519 (11)	C(12)—C(16)	1.520 (9)
C(13)—C(14)	1.537 (15)	C(14)—C(15)	1.482 (17)
C(16)—O(17)	1.240 (8)	C(16)—O(18)	1.254 (9)
C(12)—O(11)—C(15)	110.7 (7)	O(11)—C(12)—C(13)	104.8 (6)
O(11)—C(12)—C(16)	109.9 (6)	C(12)—C(12)—C(16)	113.8 (6)
C(12)—C(13)—C(14)	102.0 (7)	C(13)—C(14)—C(15)	105.2 (9)
O(11)—C(15)—C(14)	107.5 (9)	C(12)—C(16)—O(17)	119.0 (6)
C(12)—C(16)—O(18)	115.6 (6)	O(17)—C(16)—O(18)	125.4 (6)
O(18)—C(16)—C(12)—O(11)	-175.3 (7)	O(17)—C(16)—C(12)—O(11)	5.3 (3)
C(16)—C(12)—O(11)—C(15)	-147.8 (8)	C(12)—O(11)—C(15)—C(14)	7.8 (9)
O(11)—C(15)—C(14)—C(13)	12.6 (3)	C(15)—C(14)—C(13)—C(12)	-26.5 (7)
C(14)—C(13)—C(12)—C(16)	151.2 (8)	C(13)—C(12)—C(16)—O(17)	-111.9 (8)
C(13)—C(12)—C(16)—O(18)	67.5 (6)	C(13)—C(12)—O(11)—C(15)	-25.2 (6)
C(14)—C(13)—C(12)—O(11)	31.1 (6)		

Table 3. Interatomic distances ( $\text{\AA}$ ) involving water molecules

O(19)—O(1)	2.899 (7)	O(20)—O(8)	2.782 (8)
O(19)—O(7)	2.876 (8)	O(20)—O(11)	3.156 (8)
O(19)—O(18)	2.905 (8)	O(20)—O(17)	2.848 (8)
O(19)—O(18 <sup>m</sup> )	2.836 (8)	O(20)—O(7 <sup>m</sup> )	2.827 (8)
O(19)—O(17 <sup>m</sup> )	2.741 (8)	O(20)—O(8 <sup>m</sup> )	2.773 (8)

Symmetry code: (i)  $1 - x, \frac{1}{2} + y, 1 - z$ ; (ii)  $x, 1 + y, z$ ; (iii)  $x, -1 + y, z$ ; (iv)  $-x, -\frac{1}{2} + y, 1 - z$ .

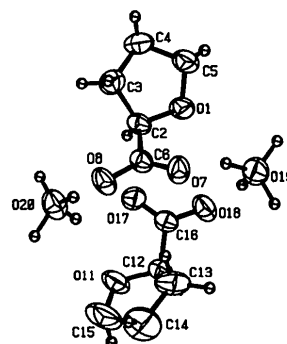


Fig. 1. ORTEP (Johnson, 1965) drawing of the two independent molecules with the atom-numbering scheme. H atoms are shown with an arbitrary thermal factor. The thermal ellipsoids correspond to 50% probability levels of atomic displacement.

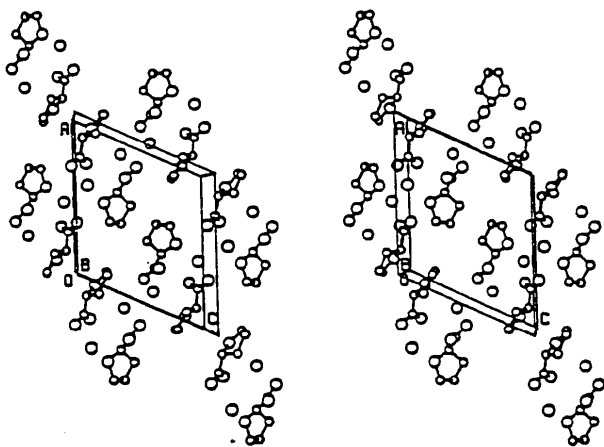


Fig. 2. Stereoscopic *PLUTO* (Motherwell, 1973) drawing of the packing of molecules viewed along the *b* axis.

Bond lengths and angles of the tetrahydrofuran rings of the molecules are similar to those of tetrahydrofuran at 148 K (Luger & Buschmann, 1983). Torsion angles indicate that the twofold ring symmetry of the latter compound no longer exists in the title compound.

The C—O bond lengths of two carboxylic groups, which are not significantly different from each other, are intermediate between single- and double-bond length. This indicates that the groups are deprotonated and electron-delocalized, though asymmetries in the C—C—O angles [C(2)—C(6)—O(7) and C(12)—C(16)—O(17) are larger than C(2)—C(6)—O(8) and C(12)—C(16)—O(18) respectively] suggest that C(6)—O(7) and C(16)—O(17) have double-bond character.

An unusual feature is the coordination of the two water molecules; four H atoms are tetrahedrally coordinated around each water O atom. All the H atoms around the water O(19) atom, and three H atoms around the water O(20) atom seem to be involved in hydrogen bonds, indicating that the water molecules take alternative orientations to form the hydrogen bonds. The packing scheme reveals that two layers of the molecules are held together by the water molecules to form a three-layered sheet. There are no interactions between the sheets except for van der Waals contacts.

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*Acta Cryst.* (1990). **C46**, 108–112

## Structures of Three *N*-Pyridyl-2-phenylsuccinimides and Structural Evidence for Substituent Effects on Anticonvulsant Properties

BY WITOLD KWIATKOWSKI AND JANINA KAROLAK-WOJCIECHOWSKA\*

*Institute of General Chemistry, Technical University, 90–924 Łódź, Zwirki 36, Poland*

AND JOLANTA OBNISKA AND ALFRED ZEJC

*Institute of Pharmaceutical Chemistry, Medical Academy, 31-065 Kraków, Skąlecka 10, Poland*

(Received 13 December 1988; accepted 2 May 1989)

**Abstract.** *T* = 295 K, Mo *K*α with  $\lambda = 0.70930 \text{ \AA}$ . Compound (I-6): C<sub>15</sub>H<sub>12</sub>N<sub>2</sub>O<sub>2</sub>, *M<sub>r</sub>* = 252.26, monoclinic, *P*2<sub>1</sub>/*c*, *a* = 8.441 (3), *b* = 15.269 (1), *c* = 9.745 (2) Å,  $\beta = 92.34 (2)^\circ$ , *V* = 1254.9 (19) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.335 g cm<sup>-3</sup>,  $\mu = 0.85 \text{ cm}^{-1}$ , *F*(000) = 528, *R* = 0.0345 for 1682 observed reflections. Compound (I-10): C<sub>16</sub>H<sub>14</sub>N<sub>2</sub>O<sub>2</sub>, *M<sub>r</sub>* = 266.30, monoclinic, *P*2<sub>1</sub>/*n*, *a* = 11.637 (1), *b* = 5.793 (1), *c* = 20.778 (2) Å,  $\beta =$

\* To whom all correspondence should be addressed.