

hydrophobic sheets (Fig. 2), each leading to a typical 'herringbone' pattern as shown in Fig. 3. Molecular modelling experiments indicate that a dimer conformation with phenyl groups oriented as shown in (I) is possible only if the P^+ side chain is in the *trans* position. This also requires a close to perpendicular orientation of the P^+ carboxyl group relative to the P carboxylate (observed interplanar angle = 27.3°). Such conformations could not support the hydrogen-bond network observed in the crystal structure.

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cis-7-Oxo-8-oxabicyclo[4.3.0]non-2-ene-5-*cis*-carboxylic Acid

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Abstract. $C_9H_{10}O_4$, $M_r = 182.15$, orthorhombic, $P2_12_12_1$, $a = 6.589$ (4), $b = 7.003$ (4), $c = 18.436$ (12) Å, $V = 850.7$ Å³, $Z = 4$, $D_x = 1.422$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.106$ mm⁻¹, $F(000) = 384$, $T = 293$ K, $R = 0.0517$ for 913 unique reflections with $F > 6\sigma(F)$. The bond lengths and angles are all within the accepted ranges. The molecule resembles a hinge with the two rings lying at approximately 110° to one another with the carboxyl group on C5 equatorial and effectively coplanar with the cyclohexene. No intramolecular

hydrogen bond is formed between O7 and O10/O11 but a network of intermolecular hydrogen bonding between carboxyl groups holds the molecules together in the *a*-axis direction. Other packing interactions are hydrophobic. The torsion angles determined by NMR are in good agreement with those determined from the crystal structure.

Experimental. The title compound was prepared by the Diels–Alder condensation of 1-hydroxypenta-2,4-diene with maleic anhydride and crystals were obtained by recrystallization from H₂O. Colourless prism, $0.4 \times 0.2 \times 0.2$ mm, mounted on Siemens–

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Table 1. Fractional coordinates of freely refined atoms, with standard deviations

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}/U_{iso}(Å²)</i>
O10	-0.0340 (4)	-0.2026 (4)	-0.50368 (15)	0.0434 (16)
O11	-0.2281 (4)	-0.2306 (5)	-0.40662 (16)	0.0481 (17)
O8	0.1575 (5)	0.3910 (4)	-0.39099 (18)	0.0560 (20)
O7	-0.1209 (5)	0.2330 (5)	-0.42321 (18)	0.0601 (21)
C10	-0.0566 (6)	-0.1883 (5)	-0.43826 (21)	0.0341 (22)
C6	0.2034 (6)	0.0669 (5)	-0.41519 (21)	0.0341 (21)
C7	0.0581 (7)	0.2324 (6)	-0.41002 (22)	0.0426 (24)
C5	0.1096 (6)	-0.1223 (6)	-0.38856 (20)	0.0363 (21)
C4	0.0524 (7)	-0.1104 (6)	-0.30803 (20)	0.0446 (23)
C9	0.3704 (7)	0.3486 (6)	-0.38190 (27)	0.055 (3)
C1	0.3844 (6)	0.1348 (6)	-0.37080 (23)	0.0402 (21)
C2	0.3676 (7)	0.0826 (6)	-0.29174 (23)	0.0466 (25)
C3	0.2180 (7)	-0.0223 (7)	-0.26451 (24)	0.0451 (24)
H11	-0.322 (8)	-0.274 (7)	-0.439 (3)	0.060 (14)

Table 2. Selected bond distances (Å), angles (°) and torsion angles (°)

O11	H11	0.95 (4)	C6	C7	1.507 (6)				
O11	C10	1.306 (5)	C6	C5	1.542 (5)				
O10	C10	1.219 (5)	C6	C1	1.522 (6)				
C10	C5	1.501 (5)	C5	C4	1.534 (6)				
C9	C1	1.514 (6)	C4	C3	1.488 (6)				
O8	C9	1.444 (6)	C2	C3	1.328 (6)				
O7	C7	1.205 (5)	C1	C2	1.507 (6)				
H11	O11	C10	113.6 (22)	C5	C6	C1	114.3 (3)		
O10	C10	O11	121.8 (3)	C10	C5	C6	111.2 (3)		
O10	C10	C5	122.7 (3)	C10	C5	C4	115.4 (3)		
O11	C10	C5	115.4 (3)	C6	C5	C4	111.1 (3)		
O8	C9	C1	106.2 (4)	C5	C4	C3	111.4 (3)		
C7	O8	C9	109.6 (3)	C4	C3	C2	124.7 (4)		
O8	C7	O7	122.0 (4)	C1	C2	C3	123.7 (4)		
O8	C7	C6	110.2 (3)	C6	C1	C9	100.9 (3)		
O7	C7	C6	127.7 (4)	C6	C1	C2	112.8 (3)		
C7	C6	C5	112.7 (3)	C9	C1	C2	111.5 (4)		
C7	C6	C1	102.9 (3)						
C6	C1	C2	C3	4.2 (6)	C1	C6	C5	C4	54.6 (4)
C9	C1	C2	C3	116.9 (5)	C7	C6	C5	C10	67.6 (4)
C1	C2	C3	C4	3.0 (7)	C1	C6	C7	O8	19.7 (4)
C5	C4	C3	C2	18.6 (6)	C1	C6	C7	O8	19.7 (4)
C6	C5	C4	C3	-45.5 (4)	C7	O8	C9	C1	-20.0 (5)
C7	C6	C5	C4	-62.4 (4)	C9	O8	C7	C6	-0.1 (4)
C5	C6	C1	C2	-33.0 (5)	O8	C9	C1	C6	30.6 (4)
C7	C6	C1	C9	-29.5 (4)	O10	C10	C5	C4	179.1 (3)

Stoe AED-2 four-circle diffractometer, graphite-monochromated Mo $K\alpha$ X-radiation, cell parameters from 16 reflections at $\pm\omega$. For data collection, $\omega/2\theta$ scans, $2\theta_{\max} = 45^\circ$, $h\ 0 \rightarrow 7$, $k\ 0 \rightarrow 7$, $l\ -19 \rightarrow 19$, 1354 reflections collected, no significant crystal movement or decay, semi-empirical absorption correction [North, Phillips & Mathews (1968); max. and min. transmission factors 0.638 and 0.395, respectively] after merging, 916 reflections with $I > 3\sigma(I)$ and $R_{\text{int}} = 0.045$. Direct methods (Sheldrick, 1986) revealed all non-H atoms, H atoms refined riding on atoms to which they were attached except H11 which was refined freely [full-matrix least squares on F (SHELX76; Sheldrick, 1976)]. Anisotropic C, O, isotropic H. At final convergence, $R = 0.0517$, $wR = 0.0655$, $S = 0.850$ for 132 parameters, $(\Delta/\sigma)_{\max}$ in the

final cycle 0.009, max. and min. residuals in final ΔF synthesis 0.28, $-0.47\ e\ \text{\AA}^{-3}$. The weighting scheme $w^{-1} = \sigma^2(F) + 0.004966F^2$ gave satisfactory agreement analyses. 3 reflections with $F_{\text{calc}} \geq F_{\text{obs}}$ ignored. Scattering factors were inlaid (Sheldrick, 1976). Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1 and bond lengths and angles appear in Table 2.* The atomic numbering scheme is shown in Fig. 1 produced by an interactive version of ORTEP (Johnson, 1965; Mallinson & Muir, 1985). Molecular geometry calculations were performed using CALC (Gould & Taylor, 1983).

Related literature. This work arises from synthetic approaches to oxa-bicyclooctane ring systems related to prostaglandin H₂ and thromboxane A₂ (Muir *et al.*, 1991). The structure here confirms the synthetic data of Heilbron, Jones, McCombie & Weedon (1945), although it is still uncertain whether esterification precedes, or is concomitant with, cycloaddition and, further, shows why subsequent halolactonization between C3 and O11 is impossible.

* Lists of torsion angles, structure factors, anisotropic thermal parameters and H-atom coordinates, together with details of the NMR analysis, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54893 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

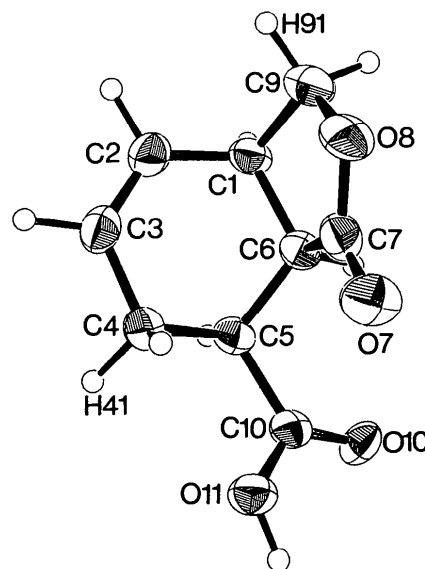


Fig. 1. A general view of the molecule showing the numbering scheme; the H atoms are numbered according to the atoms to which they are attached. Thermal ellipsoids of non-H atoms are drawn at the 50% probability level. H atom radii are 0.15 Å. In the crystal, a hydrogen bond is formed between O11—H11 and O10 of a neighbouring molecule producing a chain in the *a*-axis direction.

The synthesis produces a racemic mixture as does the similar synthesis of (±)-nor-sterepolide (Arai, Takeda, Masuda & Koizumi, 1985).

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Structure du 6-(4-Méthylpipérazin-1-yl)-11H-pyrido[2,3-b][1,4]benzodiazépine 1,5-Hydrate

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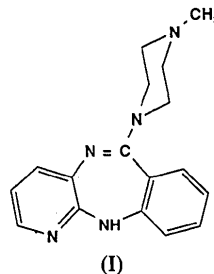
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Abstract. 6-(4-Methylpiperazin-1-yl)-11H-pyrido[2,3-b][1,4]benzodiazepine 1.5-hydrate, C₁₇H₁₉N₅·1.5H₂O, *M_r* = 320.4, triclinic, *P* $\bar{1}$, *a* = 11.268 (3), *b* = 12.815 (3), *c* = 13.720 (4) Å, α = 107.37 (1), β = 98.15 (2), γ = 110.42 (1)°, *V* = 1703.8 (1) Å³, *Z* = 4, *D_x* = 1.249 Mg m⁻³, m.p. 414 K, $\lambda(\text{Cu K}\alpha)$ = 1.5418 Å, μ = 0.592 mm⁻¹, *F*(000) = 684, *T* = 293 (1) K, *R* = 0.049 for 3510 independent reflections. The diazepine ring is in a boat conformation. The dihedral angles between the two aromatic rings in the two independent molecules are 131.7 (1) and 120.5 (1)°, respectively. The distances between the N (methylpiperazine) atom and the centres of the two aromatic rings are respectively 6.119 (4) and 7.707 (4) Å in molecule *A*, and 6.074 (4) and 7.746 (4) Å in molecule *B*. All H atoms of water molecules participate in O···O and O···N hydrogen bonds [distance ranges 2.796 (3)–2.887 (3) and 2.843 (3)–2.956 (3) Å, respectively]. Two intermolecular N···N contacts of 2.970 (4) and 3.209 (4) Å may also be considered as hydrogen bonds.

Partie expérimentale. Cristal du composé (I) incolore 0,13 × 0,22 × 0,54 mm obtenu par cristallisation

dans un mélange méthanol (50% en volume)–acétone.



Paramètres de la maille déterminés à partir de 37 réflexions telles que $17,5 \leq \theta \leq 27,4^\circ$. Balayage ω , $7,0 \leq 2\theta \leq 110,0^\circ$, $-11 \leq h \leq 11$, $-13 \leq k \leq 13$, $0 \leq l \leq 14$. Réflexions de contrôle de l'intensité: $947 \leq F_o(122) \leq 986$ et $2443 \leq F_o(225) \leq 2532$. Diffractomètre Siemens. 4509 réflexions mesurées, 4274 réflexions uniques ($R_{int} = 0,02$). Corrections de Lorentz, de polarisation, de décroissance et d'absorption [par méthode semi-empirique, analogue à celle de North, Phillips & Mathews (1968)]. Facteurs de transmission minimum et maximum: 0,94 et 1,00.