Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71044 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: LI1036]

References

Albinati, A. & Marcon, M. G. (1981). *Acta Cryst.* B**37**, 2090–2092.
Konjevic, R. (1988). *Spectrosc. Lett.* **21**, 341–347.
Siemens Crystallographic Research Systems (1990). *SHELXTL/PC*. Release 4.1. Siemens Crystallographic Research Systems, Madison, Wisconsin, USA.
Ugozzoli, F. (1987). *Comput. Chem.* **11**, 109–120.

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A Twisted Pentacyclic Diol Derviative

Barry A. Lloyd*

Department of Chemistry, Weber State University, Ogden, Utah 84408, USA

Atta M. Arif, Evan L. Allred† and Thomas R. Sharp

Department of Chemistry, University of Utah, Salt Lake City, Utah, 84112 USA

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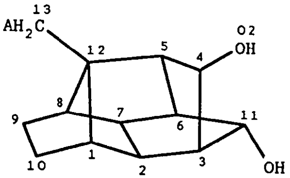
Abstract

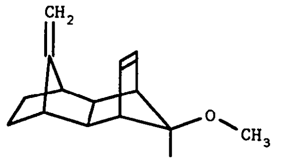
The X-ray crystal structure was determined for (*exo*,*anti*)-9-methylpentacyclo[6.3.1.$0^{3,10}$.$0^{4,12}$.$0^{5,9}$]dodecane-2,11-diol (1). The molecule consists of a twisted pentacyclic hydrocarbon ring system with a methyl substituent and two hydroxyl functional groups. Despite the two hydroxyl groups, there is no evidence of intramolecular hydrogen bonding.

Comment

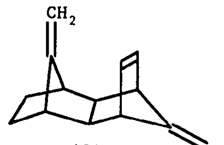
Attempted hydrolysis of the dimethoxyketal in (2) with aqueous strong acids resulted in the formation of mainly ring-closed twisted products (Lloyd, Arif, Allred, Patton & Sharp, 1993) rather than the ketone (3). However, (3) can be isolated as the major product from deuterolysis of (2) with the weakly acidic salt 2,6-lutidinium tosylate-*d* in D_2O. Ketone (3) can be reduced to the *anti*-alcohol (4) whose diene system, like that of (2) and (3), is acid sensitive. Treatment of (4) with strong aqueous acid results in the formation of the racemic ring-closed twisted product (1), the title compound.



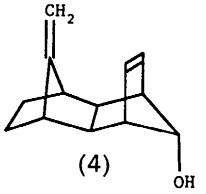
(1) A = H
(1*d*) A = D



(2)



(3)



(4)

There are three intermolecular contacts involving oxygen: $O(1^{i})\cdots O(1^{ii})$, $O(1^{i})\cdots O(2^{ii})$ and $O(1^{i})\cdots O(2^{ii})$, of 2.685 (7), 2.839 (4) and 3.089 (5) Å, respectively, which are near the sum of the van der Waals radii, 2.8 Å (Bondi, 1964). These data and angles $O(1^{i})—H(O1^{i})\cdots O(2^{ii})$ and $O(1^{i})\cdots H(O2^{ii})—O(2^{ii})$ (157.3 and 150.6°, respectively) are consistent with intermolecular hydrogen bonding. There is no evidence for intramolecular hydrogen bonding, as expected with the *exo*- and *anti*-hydroxyl group geometries. There were no other non-hydrogen intermolecular contacts ≤ 3.5 Å.

Bond lengths and bond angles are similar to those of a previously reported derivative having the same pentacyclic ring structure (Lloyd, Arif, Allred, Patton & Sharp, 1993). The two longest bonds, C(2)—C(7) and C(5)—C(6), are also consistent with those of other derivatives (Astin, Fletcher, Mackenzie, Miller, Ratcliffe, Frew & Muir, 1982; Khan, Bauer & Khan, 1972), but the C(6)—C(7) bond is not as short as those found in these structures.

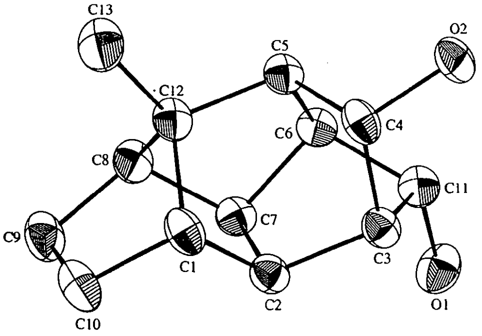


Fig. 1. Drawing of non-H atoms of one of the enantiomers of (1) with thermal ellipsoids at the 40% probability level.

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† Deceased 8 July 1991.

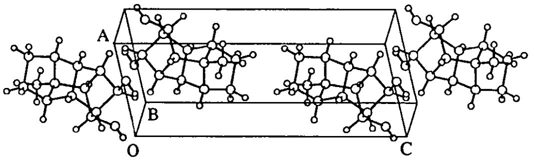

Fig. 2. Cell packing diagram of (1).

Experimental

Crystal data

$C_{13}H_{18}O_2$
$M_r = 206.29$
Triclinic
$P\bar{1}$
$a = 6.421\ (2)$ Å
$b = 6.658\ (4)$ Å
$c = 13.758\ (5)$ Å
$\alpha = 85.53\ (3)°$
$\beta = 99.70\ (3)°$
$\gamma = 117.54\ (3)°$
$V = 514.12$ Å^3
$Z = 2$

$D_x = 1.332$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 7.5$-$15°$
$\mu = 0.0820$ mm^{-1}
$T = 296$ K
Prism
0.25 × 0.23 × 0.13 mm
Colourless

Data collection

Syntex $P\bar{1}$ diffractometer
$\theta/2\theta$ scans of variable speed 3.0-8.0° min^{-1}
Absorption correction: none
1773 measured reflections
1605 independent reflections
1256 observed reflections $[I > 3\sigma(I)]$

$R_{\text{int}} = 0.0093$
$\theta_{\max} = 23°$
$h = 0 \rightarrow 7$
$k = -7 \rightarrow 7$
$l = -14 \rightarrow 14$
2 standard reflections monitored every 98 reflections intensity variation: <3%

Refinement

Refinement on F
Final $R = 0.0531$
$wR = 0.0540$
$S = 0.5522$
1256 reflections
190 parameters
Only coordinates of H atoms refined
Unit weights applied

$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.336$ e Å^{-3}
$\Delta\rho_{\min} = -0.186$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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

$$B_{\text{eq}} = \frac{8\pi^2}{3}\textstyle\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
O(1)	0.7182 (3)	0.1336 (3)	0.0469 (2)	3.39 (5)
O(2)	0.9543 (4)	0.8165 (3)	0.0796 (2)	3.27 (5)
C(1)	0.8189 (5)	0.4428 (5)	0.3316 (2)	2.87 (7)
C(2)	0.8189 (5)	0.3058 (5)	0.2465 (2)	2.52 (7)
C(3)	0.9337 (5)	0.4640 (5)	0.1593 (2)	2.49 (7)
C(4)	0.9048 (5)	0.6790 (5)	0.1663 (2)	2.69 (7)
C(5)	0.6390 (5)	0.5638 (4)	0.1748 (2)	2.48 (7)
C(6)	0.5396 (5)	0.3316 (4)	0.1179 (2)	2.36 (6)
C(7)	0.5431 (5)	0.1809 (4)	0.2068 (2)	2.40 (6)
C(8)	0.4265 (5)	0.2408 (4)	0.2796 (2)	2.71 (7)
C(9)	0.4565 (6)	0.1479 (5)	0.3830 (2)	3.53 (8)
C(10)	0.7255 (6)	0.2845 (5)	0.4188 (2)	3.76 (9)
C(11)	0.7456 (5)	0.3526 (4)	0.0700 (2)	2.62 (7)
C(12)	0.6125 (5)	0.4946 (4)	0.2847 (2)	2.59 (7)
C(13)	0.5682 (6)	0.6527 (5)	0.3429 (3)	4.10 (9)

Table 2. *Bond lengths* (Å) *and angles* (°)

O(1)—C(11)	1.441 (5)	C(5)—C(6)	1.587 (5)
O(2)—C(4)	1.432 (5)	C(5)—C(12)	1.560 (6)
C(1)—C(2)	1.539 (6)	C(6)—C(7)	1.525 (6)
C(1)—C(10)	1.540 (6)	C(6)—C(11)	1.520 (6)
C(1)—C(12)	1.547 (6)	C(7)—C(8)	1.520 (6)
C(2)—C(3)	1.573 (6)	C(8)—C(9)	1.525 (6)
C(2)—C(7)	1.584 (6)	C(8)—C(12)	1.553 (5)
C(3)—C(4)	1.538 (6)	C(9)—C(10)	1.544 (7)
C(3)—C(11)	1.527 (6)	C(12)—C(13)	1.522 (6)
C(4)—C(5)	1.536 (6)		
C(2)—C(1)—C(10)	109.8 (4)	C(2)—C(7)—C(6)	98.5 (3)
C(2)—C(1)—C(12)	98.8 (3)	C(2)—C(7)—C(8)	105.3 (3)
C(10)—C(1)—C(12)	104.7 (4)	C(6)—C(7)—C(8)	104.2 (3)
C(1)—C(2)—C(3)	111.5 (3)	C(7)—C(8)—C(9)	113.6 (4)
C(1)—C(2)—C(7)	99.8 (3)	C(7)—C(8)—C(12)	93.8 (3)
C(3)—C(2)—C(7)	104.8 (3)	C(9)—C(8)—C(12)	105.4 (4)
C(2)—C(3)—C(4)	106.4 (3)	C(8)—C(9)—C(10)	103.0 (4)
C(2)—C(3)—C(11)	102.5 (3)	C(1)—C(10)—C(9)	103.7 (4)
C(4)—C(3)—C(11)	99.3 (3)	O(1)—C(11)—C(3)	113.3 (3)
O(2)—C(4)—C(3)	112.3 (4)	O(1)—C(11)—C(6)	111.4 (3)
O(2)—C(4)—C(5)	111.8 (4)	C(3)—C(11)—C(6)	94.9 (3)
C(3)—C(4)—C(5)	98.1 (3)	C(1)—C(12)—C(5)	108.9 (3)
C(4)—C(5)—C(6)	103.2 (3)	C(1)—C(12)—C(8)	93.3 (3)
C(4)—C(5)—C(12)	108.0 (3)	C(1)—C(12)—C(13)	116.0 (4)
C(6)—C(5)—C(12)	104.9 (3)	C(5)—C(12)—C(8)	104.2 (3)
C(5)—C(6)—C(7)	98.2 (3)	C(5)—C(12)—C(13)	114.6 (4)
C(5)—C(6)—C(11)	104.8 (3)	C(8)—C(12)—C(13)	117.5 (4)
C(7)—C(6)—C(11)	102.7 (3)		

The title compound (1) was purified by preparative gas chromatography (3% SE-30 on Chrom WAW); m.p. 464-465 K. ^{1}H NMR (300 MHz, acetone-d_6): δ 0.98 (*s*, 3H), 1.45-1.79 (*m*, 8H), 2.09-2.33 (*m*, 3H), 2.91-2.92 (*m*, 2H), 3.95 (*m*, 1H), 4.65 (*m*, 1H). ^{13}C[^{1}H] NMR (20 MHz, methanol-d_4): δ 14.89, 23.66, 26.21, 43.40, 43.69, 49.60, 50.21, 50.48, 51.11, 53.90, 55.28, 72.03, 76.40. IR (KBr pellet): 3600-3100 (*s*), 3000-2850 (*s*), 1378 (*w*), 1100-1040 (*s*) cm^{-1}. High-resolution mass spectrum of monodeuterated (1*d*) from the reaction of (4) in D_2SO_4/D_2O, *m*/*z* (relative intensity): 207.1324 (2.5) (Lloyd, 1985). Exact mass calculated for $^{12}C_{13}{}^1H_{17}{}^2H^{16}O_2$: 207.1370. A crystal of (1) suitable for X-ray structure analysis was cleaved from a larger crystal obtained by slow evaporation from acetone at room temperature. Computations were performed using the Enraf-Nonius (1979) *Structure Determination Package.*

The authors would like to thank WSU Research and Professional Growth and University of Utah University Research Committees for supporting this work.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry, details of the synthesis of (1), (3) and (4), and spectral data for (3) and (4) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71045 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1010]

References

Astin, K. B., Fletcher, A. V., Mackenzie, K., Miller, A. S., Ratcliffe, N. M., Frew, A. A. & Muir, K. W. (1982). *J. Chem. Soc. Perkin Trans.* 2, pp. 111-124.

Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
Enraf–Nonius (1979). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.
Khan, A. A., Bauer, W. H. & Khan, M. A. Q. (1972). *Acta Cryst.* **B28**, 2060–2065.
Lloyd, B. A. (1985). PhD Dissertation. Univ. of Utah, USA.
Lloyd, B. A., Arif, A. M., Allred, E. L., Patton, A. & Sharp, T. R. (1993). *Acta Cryst.* **C49**, 261–263.

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Structure of 3-(3,5-Dimethylpiperidino)-*N*-(*p*-chlorophenyl)succinimide

V. A. IGONIN, S. V. LINDEMAN* AND
YU. T. STRUCHKOV

*Nesmeyanov Institute of Organoelement Compounds,
Russian Academy of Sciences, 28 Vavilov St.,
Moscow B-334, Russia*

E. HUIPE N. AND V. MENDOZA

*Istituto Tecnologico de Morelia, Apdo. Postal 13-G,
Morelia Mich., Mexico*

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Abstract

In the title molecule, 3-(3,5-dimethylpiperidino)-1-(4-chlorophenyl)-2,5-pyrrolidinedione (1), the *N*-(*p*-chlorophenyl) substituent is rotated by 68.8° relative to the succinimide plane. The piperidinyl ring has a chair conformation with all substituents in equatorial positions; the conformation around the piperidino–succinimide C—N bond is staggered.

Comment

The title compound was prepared in the framework of our structural and conformational studies (Joseph-Nathan, Mendoza & Garcia G., 1972, 1974; Soriano-García, Toscano, Mendoza, García G., Guzmán, Alemán & Huipe N., 1990; Mendoza, Garcia G., Guzmán, Gutierrez & Chavez, 1991) of some 3-(1-cycloalkylamine)succinimides derived by condensation reactions from maleimides or isomaleimides and cycloalkylamines.

The chemical structure of (1) as well as its molecular stereochemistry are unequivocally established by the present X-ray investigation. The central succinimide moiety is planar within 0.004 Å. Its N atom [N(2)] has a planar-trigonal bond configuration with the *N*-(*p*-chlorophenyl) substituent, rotated by 68.8° around the C(10)—N(2) bond, out of the succinimide plane. The dimethylpiperidine substituent has a chair conformation with both methyl groups and the succinimide moiety in equatorial positions. The conformation around the N(1)—C(8) bond is staggered; all relevant torsion angles are close to 60 and 180°. The piperidine N(1) atom has a trigonal-pyramidal bond configuration; its displacement from the plane of three neighbouring atoms is 0.422 (4) Å.

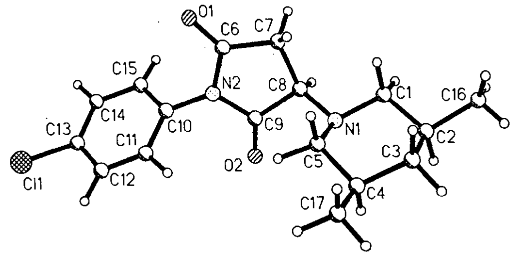

Fig. 1. General view of the title molecule (1).

Experimental

Crystal data

$C_{17}H_{21}ClN_2O_2$
$M_r = 320.8$
Monoclinic
$P2_1/c$
$a = 8.804$ (3) Å
$b = 6.472$ (2) Å
$c = 29.075$ (12) Å
$\beta = 94.75$ (2)°
$V = 1651.0$ (3) Å^3
$Z = 4$
$D_x = 1.291$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 24 reflections
$\theta = 12$–13°
$\mu = 0.240$ mm^{-1}
$T = 153$ K
Needles
0.4 × 0.2 × 0.1 mm
Colourless
Crystal source: from hexane–ethyl acetate solution (2/3)

Data collection

Siemens *P*3/*PC* diffractometer
$\theta/2\theta$ scans
Absorption correction: none
6095 measured reflections
5773 independent reflections
1176 observed reflections [$F > 6.0\sigma(F)$]

$R_{\rm int} = 0.049$
$\theta_{\rm max} = 30$°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 9$
$l = -42 \rightarrow 43$
2 standard reflections monitored every 98 reflections intensity variation: ±1.8%

Refinement

Refinement on F
Final $R = 0.052$
$wR = 0.064$
$S = 2.16$
1176 reflections
262 parameters
H atoms refined isotropically with fixed $U_{\rm iso} = 0.05$ Å^2
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\rm max} = 0.333$
$\Delta\rho_{\rm max} = 0.40$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.29$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

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