

Fig. 2. A stereoview of the unit cell showing hydrogen bonds.

to the pyridine ring in molecule *A* [torsion angle C(6)N(2)—C(1)C(2) is 167.9 (2) in molecule *A* and -3.4 (3)° in molecule *B*]. The benzyl group in molecule *B* is also rotated around the N(2)—C(10) bond resulting in a rather extended conformation of the two six-membered rings as compared with the conformation of molecule *A* wherein the two rings are *cis* about the N(2)—C(10) bond [torsion angle C(1)N(2)—C(10)—C(11) is 93.3 (2) and -91.2 (2)° in molecules *A* and *B*, respectively].

Bond lengths and angles in the two molecules (Table 2) are normal. There are, however, slight differences in the bond lengths and angles between the two independent molecules. For example, the N(1)—C(5) bond in molecule *B* [1.355 (3) Å] is significantly longer than the N—C bonds in the pyridine rings of the two molecules, which range between 1.341 (2) and 1.344 (2) Å. This significantly long bond in molecule *B* is matched by a similar lengthening of the C(1)—C(2) bond in molecule *A* [1.379 (4) Å]; the rest of the C—C bonds in the pyridine moieties of the two molecules range between 1.349 (2) and 1.358 (3) Å. The N(2)—C(10) bond in molecule *B* [1.455 (2) Å] is 0.013 Å longer than the

corresponding bond in molecule *A* which could be a consequence of the conformation adopted by molecule *B* about this bond; the strong interactions thus originating from this conformation seem to influence the C(14)—C(15) bond length in molecule *B* which is significantly shorter than the C(14)—C(15) bond in molecule *A*. The effects of different conformations are also manifested in small but significant differences in angles between the two molecules, *e.g.* the angle C(1)—N(2)—C(10) is 122.4 (1) and 120.3 (1)° in molecules *A* and *B*, respectively.

The phenyl and pyridine rings are planar to within 0.011 (2) Å. Each of the cations contains one H atom, H(N3), on the dimethylamino group which forms a relatively strong hydrogen bond to the Cl[−] ion. The N...Cl distances are 3.027 (1) and 2.992 (1) Å, the H(N3)...Cl distances are 2.11 (2) and 2.09 (2) Å, and the Cl...H(N3)...N(3) angles are 177 (1) and 174 (1)° for cations *A* and *B*, respectively. There are no unusual intermolecular distances less than van der Waals distances and the crystal appears to be composed of hydrogen-bonded cation–anion species (Fig. 2).

References

- B. A. FRENZ & ASSOCIATES INC. (1985). *Enraf–Nonius Structure Determination Package*. College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.
- CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- KLYNE, W. & PRELOG, V. (1960). *Experientia*, **16**, 521–523.
- NORTH, A. C. T., PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
- WILHELM, R. E. (1961). *Med. Clin. N. Am.* **45**, 887–906.
- WITAK, D. T. (1970). *Medicinal Chemistry*, edited by A. BURGER, pp. 1643–1668. New York: John Wiley.

Acta Cryst. (1987). **C43**, 1410–1412

Structure of 1-(1-Hydroxy-1-methylethyl)-1-vinyl-3,5-dioxatricyclo[5.4.1.0^{2,6}]dodeca-8,10-dien-12-ol

BY MASOOD PARVEZ, KEN S. FELDMAN AND BENEDICT J. KOSMIDER

Department of Chemistry, Pennsylvania State University, University Park, PA 16802, USA

(Received 10 September 1986; accepted 3 March 1987)

Abstract. C₁₇H₂₄O₄, *M*_r = 292.38, monoclinic, *P*2₁/*n*, *a* = 16.499 (4), *b* = 8.422 (5), *c* = 11.173 (2) Å, β = 93.96 (2)°, *V* = 1548.9 Å³, *Z* = 4, *D*_x = 1.254 g cm^{−3}, λ(Mo Kα) = 0.71073 Å, μ = 0.82 cm^{−1}, *F*(000) = 632,

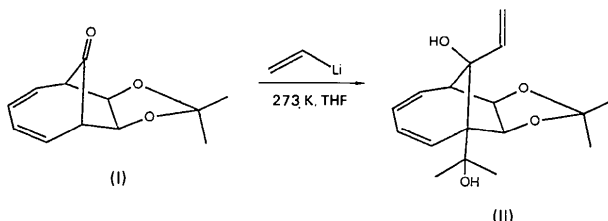
0108-2701/87/071410-03\$01.50

T = 293 K, *R* = 0.050 for 1354 observed reflections. The seven-membered ring has an envelope conformation with C(2) 0.906 (3) Å out of the plane described by the remaining atoms of the ring. The five-membered

© 1987 International Union of Crystallography

ring C(1),C(2),C(3),C(8),C(9) has a twisted envelope conformation and is inclined at $86.0(1)^\circ$ to the plane of the seven-membered ring.

Experimental. The title compound (II) was prepared by adding vinyl lithium dropwise to a stirred solution of the acetonide (I) at 273 K. Bright-orange solution quenched at 273 K with 1M H_3PO_4 , extracted with ether, washed with $NaHCO_3$ and brine, dried over Na_2SO_4 , purified by HPLC and crystallized from hexane.



Data crystal $0.24 \times 0.18 \times 0.32$ mm, Enraf-Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, cell constants and orientation matrix from 25 reflections with $10 < \theta < 20^\circ$, three standard reflections measured at regular intervals, maximum variation 1%. $\omega/2\theta$ scan method, ω -scan width $(0.60 + 0.35 \tan \theta)^\circ$, 2129 reflections (h 0 to 17, k 0 to 8, l -11 to 11) with $2 < \theta < 22^\circ$, 2057 unique, 1354 with $I > 3\sigma(I)$, L_p correction, no absorption correction.

Structure solved by direct methods, refinement by full-matrix least-squares calculations on F^2 s, non-H atoms refined anisotropically, H atoms from difference map, included at fixed positions with an overall isotropic temperature factor. $R = 0.050$, $wR = (\sum w\Delta^2 / \sum wF_o^2)^{1/2} = 0.075$, $w = [\sigma^2(F_o) + (0.08F_o)^2]^{-1}$, max. $\Delta/\sigma = 0.0$ on final cycle, $\Delta\rho$ in final ΔF map between 0.3 and -0.2 e \AA^{-3} , goodness of fit = 1.68. Scattering factors from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965). Computer programs from the Enraf-Nonius *Structure Determination Package* (B. A. Frenz & Associates Inc., 1985).

Discussion. Final fractional coordinates with equivalent isotropic temperature factors are given in Table 1. Table 2 contains molecular dimensions.* A perspective view of the molecular structure with the numbering scheme is shown in Fig. 1. The molecular dimensions are in accord with accepted values, e.g.

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, molecular dimensions involving H atoms, torsion angles and the mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43847 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters with e.s.d.'s in parentheses*

$$B_{eq} = \frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (\AA^2)
O(1)	0.7025 (1)	0.2896 (3)	0.0736 (2)	3.4 (1)
O(2)	0.6578 (1)	0.1754 (3)	-0.0987 (2)	3.9 (1)
O(3)	0.9314 (1)	0.0788 (3)	-0.0679 (2)	3.7 (1)
O(4)	0.8487 (2)	0.4253 (3)	0.1240 (2)	4.9 (1)
C(1)	0.8324 (2)	0.1361 (4)	0.0843 (3)	2.7 (1)
C(2)	0.8524 (2)	0.1375 (4)	-0.0511 (3)	2.8 (1)
C(3)	0.7842 (2)	0.0300 (4)	-0.1118 (3)	3.1 (1)
C(4)	0.8065 (2)	-0.1427 (4)	-0.1162 (3)	3.9 (1)
C(5)	0.8375 (2)	-0.2276 (4)	-0.0239 (3)	4.0 (1)
C(6)	0.8571 (2)	-0.1704 (4)	0.0966 (3)	3.8 (1)
C(7)	0.8563 (2)	-0.0242 (4)	0.1414 (3)	3.4 (1)
C(8)	0.7392 (2)	0.1354 (4)	0.0791 (3)	2.8 (1)
C(9)	0.7097 (2)	0.0577 (4)	-0.0419 (3)	3.1 (1)
C(10)	0.6331 (2)	0.2811 (4)	-0.0103 (3)	3.6 (1)
C(11)	0.5609 (2)	0.2171 (6)	0.0505 (4)	5.5 (1)
C(12)	0.6190 (3)	0.4409 (5)	-0.0667 (4)	5.4 (1)
C(13)	0.8463 (2)	0.2963 (4)	-0.1112 (3)	3.6 (1)
C(14)	0.9006 (3)	0.3585 (5)	-0.1776 (4)	5.2 (1)
C(15)	0.8731 (2)	0.2697 (4)	0.1669 (3)	3.3 (1)
C(16)	0.9645 (3)	0.2672 (6)	0.1652 (4)	5.7 (1)
C(17)	0.8466 (2)	0.2544 (5)	0.2940 (3)	4.2 (1)

Table 2. *Molecular dimensions ($\text{\AA},^\circ$) with e.s.d.'s in parentheses*

O(1)-C(8)	1.433 (4)	C(3)-C(4)	1.502 (4)
O(1)-C(10)	1.431 (3)	C(3)-C(9)	1.520 (4)
O(2)-C(9)	1.430 (3)	C(4)-C(5)	1.327 (4)
O(2)-C(10)	1.411 (3)	C(5)-C(6)	1.446 (4)
O(3)-C(2)	1.418 (4)	C(6)-C(7)	1.330 (4)
O(4)-C(15)	1.443 (3)	C(8)-C(9)	1.550 (4)
C(1)-C(2)	1.570 (4)	C(10)-C(11)	1.511 (5)
C(1)-C(7)	1.533 (4)	C(10)-C(12)	1.497 (4)
C(1)-C(8)	1.534 (4)	C(13)-C(14)	1.311 (4)
C(1)-C(15)	1.576 (4)	C(15)-C(16)	1.511 (4)
C(2)-C(3)	1.562 (4)	C(15)-C(17)	1.520 (4)
C(2)-C(13)	1.497 (4)		
O(1)...O(4)	2.695	O(1)...H(O4)	1.972
O(3)...O(3')	2.953	O(3')...H(O3)	2.362
C(8)-O(1)-C(10)	107.5 (2)	C(1)-C(7)-C(6)	131.9 (2)
C(9)-O(2)-C(10)	108.6 (2)	O(1)-C(8)-C(1)	114.7 (3)
C(2)-C(1)-C(7)	110.0 (3)	O(1)-C(8)-C(9)	103.8 (2)
C(2)-C(1)-C(8)	103.9 (2)	C(1)-C(8)-C(9)	106.8 (3)
C(2)-C(1)-C(15)	116.7 (2)	O(2)-C(9)-C(3)	111.0 (3)
C(7)-C(1)-C(8)	103.9 (2)	O(2)-C(9)-C(8)	103.7 (2)
C(8)-C(1)-C(15)	107.3 (2)	C(3)-C(9)-C(8)	107.3 (2)
C(8)-C(1)-C(15)	114.2 (2)	O(1)-C(10)-O(2)	103.5 (3)
O(3)-C(2)-C(1)	112.4 (2)	O(1)-C(10)-C(11)	110.1 (2)
O(3)-C(2)-C(3)	112.4 (2)	O(1)-C(10)-C(12)	108.9 (2)
O(3)-C(2)-C(13)	106.6 (2)	O(2)-C(10)-C(11)	111.4 (3)
C(1)-C(2)-C(3)	102.8 (3)	O(2)-C(10)-C(12)	108.4 (2)
C(1)-C(2)-C(13)	115.3 (2)	C(11)-C(10)-C(12)	114.0 (3)
C(3)-C(2)-C(13)	107.4 (3)	C(2)-C(13)-C(14)	125.7 (3)
C(2)-C(3)-C(4)	113.9 (2)	O(4)-C(15)-C(1)	111.0 (3)
C(2)-C(3)-C(9)	105.8 (3)	O(4)-C(15)-C(16)	105.3 (3)
C(4)-C(3)-C(9)	112.0 (2)	O(4)-C(15)-C(17)	107.3 (2)
C(3)-C(4)-C(5)	125.3 (3)	C(1)-C(15)-C(16)	111.6 (2)
C(4)-C(5)-C(6)	126.5 (3)	C(1)-C(15)-C(17)	110.4 (2)
C(5)-C(6)-C(7)	130.6 (3)	C(16)-C(15)-C(17)	111.2 (3)
O(3)-H(O3)...O(3')	107.2	O(4)-H(O4)...O(1)	129.7

The primed atom is related to the unprimed atom by the symmetry: $-x + 2, -y, -z$.

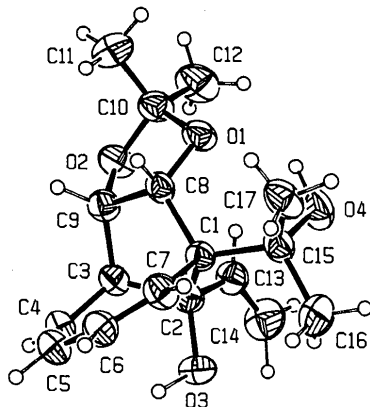


Fig. 1. Perspective drawing of the molecular structure with the crystallographic numbering scheme.

$C_{sp^3}-C_{sp^3}$ 1.497 (4)–1.576 (4) [mean 1.535 (4)], $C_{sp^3}-C_{sp^2}$ 1.497 (4)–1.533 (4) [mean 1.511 (4)], $C_{sp^2}-C_{sp^2}$ 1.446 (4), $C_{sp^2}=C_{sp^2}$ 1.311 (4)–1.330 (4) [mean 1.323 (4)], $C_{sp^3}-O$ 1.411 (3)–1.443 (3) [mean 1.426 (3)], and $C-H$ 0.941–1.206 Å. The seven-membered ring C(1)–C(7) has a twisted envelope conformation with C(2) 0.906 (3) Å out of the plane described by the remaining atoms of the ring. The five-membered ring C(1), C(2), C(3), C(8), C(9) has a C(2) envelope conformation with C(2) 0.566 (3) Å out of the best least-squares plane passing through C(1), C(3), C(8) and C(9). The five-membered ring O(1), O(2), C(8), C(9), C(10) has a C(10) envelope conformation with C(10) 0.496 (4) Å out of the plane of the remaining atoms in this ring. The five-membered rings are *cis*-fused to each other.

Acta Cryst. (1987). **C43**, 1412–1415

Structures of 3,6-Dimethoxy-1,2,4,5-tetrazine (V) and 3,6-Bis(1-aziridinyl)-1,2,4,5-tetrazine (VI)

BY CLAUS KRIEGER, HANS FISCHER AND FRANZ A. NEUGEBAUER*

Abteilung Organische Chemie, Max-Planck-Institut für medizinische Forschung, Jahnstr. 29, D-6900 Heidelberg, Federal Republic of Germany

AND FRIEDEMANN GÜCKEL AND DIETER SCHWEITZER

Abteilung für Molekulare Physik, Max-Planck-Institut für medizinische Forschung, Jahnstr. 29, D-6900 Heidelberg, Federal Republic of Germany

(Received 28 January 1987; accepted 12 March 1987)

Abstract. (V): $C_4H_6N_4O_2$, $M_r = 142.12$, orthorhombic, *Pbca*, $a = 6.865$ (3), $b = 13.192$ (5), $c =$

7.013 (3) Å, $V = 635.1$ (7) Å³, $Z = 4$, $D_x = 1.486$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.139$ cm⁻¹, $F(000) = 296$, $T = 295$ K, $R = 0.043$ for 589 unique observed reflections [$I \geq 1.96\sigma(I)$]. (VI): $C_6H_8N_6$,

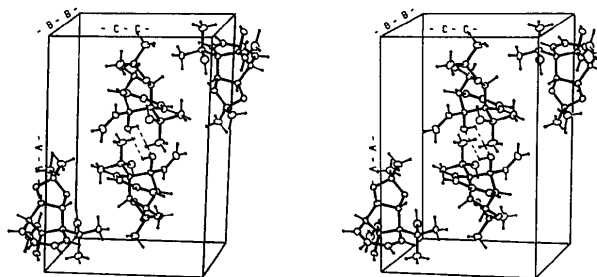


Fig. 2. A stereoview of the molecular packing diagram for (II); dashed lines indicate O–H...O hydrogen bonds.

There is a short intramolecular contact of 2.695 Å between O(1) and O(4) [O(1)...H(O4) 1.972 Å and O(4)–H(O4)...O(1) 129.7°]. The shortest intermolecular contacts in the crystal structure (Fig. 2) involve the hydroxy groups of the adjacent molecules related by an inversion center [O(3)...O(3') 2.953, O(3')...H(O3) 2.362 Å, and O(3)–H(O3)...O(3') 107.2°].

We thank the Public Health Service (1 RO1 GM35727 01A1) for financial support of this work.

References

- B. A. FRENZ & ASSOCIATES INC. (1985). *Enraf-Nonius Structure Determination Package*. College Station, Texas, USA, and Enraf-Nonius, Delft, The Netherlands.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

* To whom correspondence should be addressed.