

- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- Molecular Structure Corporation (1987). *TEXSAN, TEXRAY Structure Analysis Package*, version 2.1. Molecular Structure Corporation, College Station, Texas, USA.
- OLAH, G. A., SURYA PRAKASH, G. K., KOBAYASHI, T. & PAQUETTE, L. A. (1988). *J. Am. Chem. Soc.* **110**, 1304–1305.
- PAQUETTE, L. A. (1981). *Org. Synth. Today Tomorrow (IUPAC)*, pp. 335–347.
- PAQUETTE, L. A. (1982). *Proc. Natl Acad. Sci. USA*, **79**, 4495–4500.
- PAQUETTE, L. A. (1983). *Chem. Aust.* **50**, 138–142.
- PAQUETTE, L. A. (1984). *Strategies and Tactics of Organic Synthesis*, edited by T. LINDBERG, pp. 175–200. New York: Academic Press.
- PAQUETTE, L. A., BALOGH, D. W., USHA, R., KOUNTZ, D. & CHRISTOPH, G. G. (1981). *Science*, **211**, 575–576.
- PAQUETTE, L. A., KOBAYASHI, T. & GALLUCCI, J. C. (1988). *J. Am. Chem. Soc.* **110**, 1305–1307.
- PAQUETTE, L. A., WEBER, J. C. & KOBAYASHI, T. (1988). *J. Am. Chem. Soc.* **110**, 1303–1304.
- SANTOS, E., BALOGH, D. W., DOECKE, C. W., MARSHALL, A. G. & PAQUETTE, L. A. (1986). *J. Am. Chem. Soc.* **108**, 8183–8185.
- SCAMEHORN, C. A., HERMILLER, S. M. & PITZER, R. M. (1986). *J. Chem. Phys.* **84**, 833–837.
- SCHULMAN, J. M. & DISCH, R. L. (1978). *J. Am. Chem. Soc.* **100**, 5677–5681.
- SCHULMAN, J. M. & DISCH, R. L. (1984). *J. Am. Chem. Soc.* **106**, 1202–1204.
- SCHULMAN, J. M., VENANZI, T. & DISCH, R. L. (1975). *J. Am. Chem. Soc.* **97**, 5335–5339.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

*Acta Cryst.* (1989). **C45**, 898–900

### Structure of 3,3a,5,6,7,8b-Hexahydro-2H,8H-furo[3,2-b]benzofuran-2,8-dione\*

BY M. SORIANO-GARCÍA,† F. WALLS, H. BARRIOS, B. ORTÍZ, R. SÁNCHEZ-OBREGÓN AND F. YUSTE

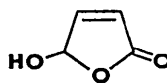
*Instituto de Química, Universidad Nacional Autónoma de México, Circuito Exterior, Ciudad Universitaria, Coyoacán 04510, Mexico DF*

(Received 16 June 1988; accepted 17 November 1988)

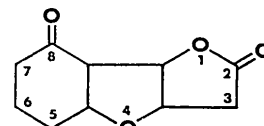
**Abstract.** C<sub>10</sub>H<sub>10</sub>O<sub>4</sub>, *M<sub>r</sub>* = 194.2, monoclinic, *P*2<sub>1</sub>/*c*, *a* = 5.442 (1), *b* = 18.162 (6), *c* = 9.220 (3) Å, β = 105.73 (2)°, *V* = 877 (1) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.47 Mg m<sup>-3</sup>, λ(Mo Kα) = 0.7107 Å, μ = 0.107 mm<sup>-1</sup>, *F*(000) = 408, *T* = 293 K, *R* = 0.046 for 1167 observed reflections. The X-ray study confirms that the solid state structure is similar to that inferred from chemical and spectroscopic evidence. The six-membered *A* ring adopts a 1,2-diplanar conformation whilst both five-membered rings (*B* and *C*) are intermediate between half-chair and β-envelope. The *A/B* and *B/C* junctions are *cis*. The packing in the crystal is entirely due to intermolecular C–H⋯O contacts and van der Waals forces.

**Introduction.** The base-catalyzed reaction of 5-hydroxy-2(5*H*)-furanone (1) with several cyclic and acyclic vinylogous amides and 1,3-dicarbonyl compounds was found to provide a smooth method of preparing pyrrole, furan, tetrahydroindole and tetrahydrobenzofuran ring systems (Yuste & Sánchez-Obregón, 1982). The title compound (2) has been synthesized. The empirical formula C<sub>10</sub>H<sub>10</sub>O<sub>4</sub> was established by mass spectrometry and the functional

groups present were characterized by NMR, IR and UV spectroscopy (Yuste & Sánchez-Obregón, 1982). We have undertaken the X-ray study of (2).



(1)



(2)

**Experimental.** Compound (2) was obtained from the reaction of 1.12 g (10 mmol) of 1,3-cyclohexanedione, 1 g (10 mmol) of lactone (1) and 1 ml of 2% sodium hydroxide in 30 ml of ethanol, by refluxing for 2 h. The product crystallized on cooling and subsequent recrystallization from cold ethanol gave colourless crystals, m.p. 430–431 K. Size of crystal 0.14 × 0.14 × 0.42 mm. Nicolet R3 four-circle diffractometer. Unit-cell parameters by least squares from 25 machine-centred reflections with 4.5 < 2θ < 16.5°. 1551 unique reflections measured for two octants, 3 < 2θ < 50°, 1167 with *I* > 2.5σ(*I*) used in analysis, index range *h* ±6, *k* 0→21, *l* 0→10, *R<sub>int</sub>* = 0.021, 2θ/θ scan mode, variable scan speed, scan width 1.0° (θ). Two standard reflections (131, 130) monitored every 50 measurements, no significant variation. Intensities

\* Contribution No. 956 of the Instituto de Química, UNAM.

† To whom correspondence should be addressed.

corrected for Lorentz–polarization but not for absorption. Data adjusted to an approximately absolute scale, overall  $U = 0.047 \text{ \AA}^2$ . Structure solved by combination of direct methods and partial structure expansion by an iterative *E*-Fourier procedure using *SHELXTL5* (Sheldrick, 1985). Least-squares refinement with all non-H atoms treated anisotropically; H atoms in calculated positions riding on bonded C with a fixed isotropic temperature factor,  $U = 0.06 \text{ \AA}^2$ .  $\sum w(\Delta F)^2$  minimized,  $w = [\sigma^2(F_o) + 0.00109(F_o)^2]^{-1}$ , where  $\sigma$  is standard deviation of observed amplitudes, based on counting statistics; isotropic extinction parameter  $X = 0.004$ . In the last cycle  $(\Delta/\sigma)_{\max} = 0.14$ ;  $\Delta\rho$  from  $-0.19$  to  $0.17 \text{ e \AA}^{-3}$ ,  $S = 1.18$ ; final  $R = 0.046$ ,  $wR = 0.054$ ; scattering factors from *International Tables for X-ray Crystallography* (1974). All computations performed on a Nova 4S computer and plots drawn on a Tektronix plotter with the *SHELXTL* system of programs.

**Discussion.** Atomic coordinates are given in Table 1.\* Fig. 1 shows the conformation of (2). The bond lengths and the valence angles for non-H atoms are listed in Table 2. The molecular structure of (2) determined from X-ray data confirms the structure previously assigned on the basis of chemical and spectroscopic methods.

The conformation of ring *A* may be described as 1,2-diplanar, with C(6)  $-0.60(7) \text{ \AA}$  out of the best plane formed by the other five ring atoms. The *B* and *C* rings have  $\Delta$  and  $\varphi_m$  values (Altona, Geise & Romers, 1968) of  $16.7(2)$ ,  $8.5(2)$  and  $20.2(3)$ ,  $9.4(3)^\circ$  indicating, in both cases, an intermediate between a half-chair and a  $\beta$ -envelope conformation. At the *A/B* and *B/C* *cis* junctions the torsion angles C(5)–C(4a)–C(8a)–C(8), O(4)–C(4a)–C(8a)–C(8b) and O(4)–C(3a)–C(8b)–C(8a), C(3)–C(3a)–C(8b)–O(1) are  $-4.4(4)$ ,  $-1.7(3)$  and  $-8.4(2)$ ,  $-8.7(2)^\circ$ . The angles between the best planes through the *A/B*, *B/C* and *A/C* rings are  $6.7(6)$ ,  $117.0(7)$  and  $112.8(7)^\circ$ , respectively.

The carbonyl groups at C(2) and C(8) have C(2)–O(2) =  $1.198(4)$  and C(8)–O(3) =  $1.223(3) \text{ \AA}$ , agreeing well with the accepted value of  $1.215(5) \text{ \AA}$  (Sutton, 1965). Internal ring angles at these carbonyl C atoms are O(1)–C(2)–C(3)  $111.1(2)$  and C(7)–C(8)–C(8a)  $114.6(2)^\circ$ , respectively.

In the *B* and *C* rings, there is an asymmetry in the C–O endocyclic bond lengths [C(2)–O(1)  $1.349(3)$ , C(8b)–O(1)  $1.465(3)$  and C(3a)–O(4)  $1.472(3)$ ,

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic temperature factors ( $\text{\AA}^2 \times 10^3$ )

$$U_{eq} = (U_{11} \times U_{22} \times U_{33})^{1/3}$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}$
O(1)	1348(3)	6650(1)	2983(2)	54(1)
C(2)	2912(5)	6198(2)	3979(3)	51(1)
C(3)	5356(5)	6080(1)	3578(2)	41(1)
C(3a)	4989(4)	6449(1)	2064(2)	36(1)
O(4)	4565(3)	5901(1)	846(2)	41(1)
C(4a)	2298(4)	6051(1)	-134(2)	33(1)
C(5)	1445(5)	5602(1)	-1526(3)	44(1)
C(6)	-1402(5)	5694(1)	-2186(3)	47(1)
C(7)	-2231(5)	6498(1)	-2203(3)	44(1)
C(8)	-1413(4)	6861(1)	-675(2)	35(1)
C(8a)	990(4)	6603(1)	280(2)	32(1)
C(8b)	2486(4)	6877(1)	1785(2)	37(1)
O(2)	2297(4)	5949(2)	5033(2)	89(1)
O(3)	-2671(3)	7348(1)	-302(2)	48(1)

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

O(1)–C(2)	1.349(3)	O(1)–C(8b)	1.465(3)
C(2)–C(3)	1.489(4)	C(2)–O(2)	1.198(4)
C(3)–C(3a)	1.512(3)	C(3a)–O(4)	1.472(3)
C(3a)–C(8b)	1.529(3)	O(4)–C(4a)	1.344(2)
C(4a)–C(5)	1.486(3)	C(4a)–C(8a)	1.343(3)
C(5)–C(6)	1.512(3)	C(6)–C(7)	1.527(3)
C(7)–C(8)	1.510(3)	C(8)–C(8a)	1.442(3)
C(8)–O(3)	1.223(3)	C(8a)–C(8b)	1.492(3)
C(2)–O(1)–C(8b)	111.5(2)	O(1)–C(2)–O(2)	121.2(3)
O(1)–C(2)–C(3)	111.1(2)	C(3)–C(2)–O(2)	127.7(2)
C(2)–C(3)–C(3a)	105.2(2)	C(3)–C(3a)–C(8b)	105.7(2)
C(3)–C(3a)–O(4)	111.1(2)	O(4)–C(3a)–C(8b)	105.5(2)
C(3a)–O(4)–C(4a)	107.6(2)	O(4)–C(4a)–C(8a)	114.9(2)
O(4)–C(4a)–C(5)	119.0(2)	C(5)–C(4a)–C(8a)	126.1(2)
C(4a)–C(5)–C(6)	109.5(2)	C(5)–C(6)–C(7)	112.2(2)
C(6)–C(7)–C(8)	113.4(2)	C(7)–C(8)–O(3)	122.2(2)
C(7)–C(8)–C(8a)	114.6(2)	C(8a)–C(8)–O(3)	123.2(2)
C(4a)–C(8a)–C(8)	122.2(2)	C(8)–C(8a)–C(8b)	129.4(2)
C(4a)–C(8a)–C(8b)	108.4(2)	C(3a)–C(8b)–C(8a)	102.9(2)
O(1)–C(8b)–C(8a)	111.8(2)	O(1)–C(8b)–C(3a)	105.6(2)

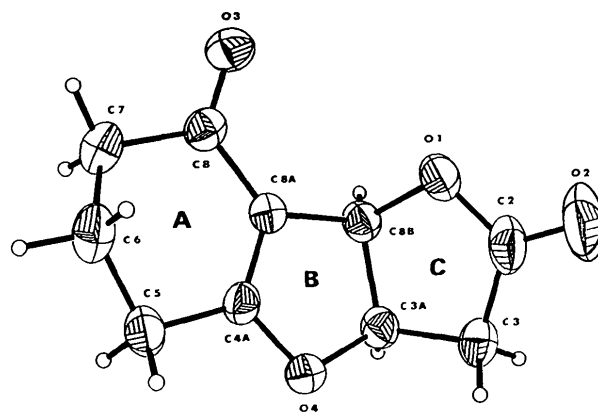


Fig. 1. The molecular conformation of (2), showing the atom numbering. The thermal ellipsoids are drawn at the 50% probability level.

C(4a)–O(4)  $1.344(2) \text{ \AA}$ ]; the contraction of the C(2)–O(1) and C(4a)–O(4) single bonds is typical of  $-\text{O}-\text{C} \text{ sp}^2$  substructures.

\* Lists of structure amplitudes, anisotropic thermal parameters, H-atom coordinates and the results of mean-plane calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51623 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

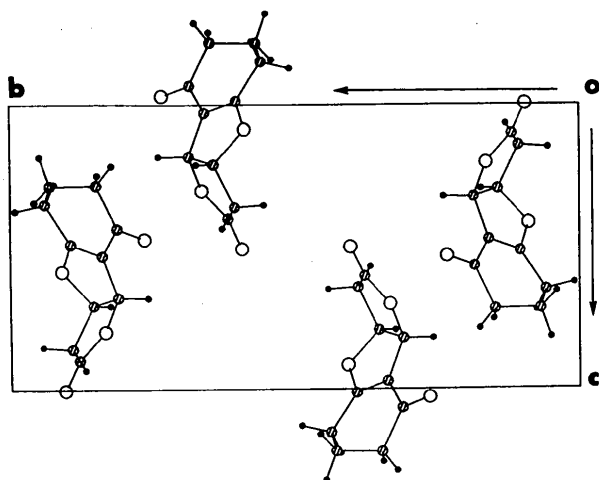


Fig. 2. Packing arrangement as viewed along *a*.

Examination of the three-dimensional packing of the molecules within the crystalline lattice (shown in Fig. 2) reveals that, excluding H atoms, there are five intermolecular approaches  $< 3.4 \text{ \AA}$ : C(3a)···O(1), C(3a)···O(3),  $(1+x, 1.5-y, 0.5+z)$  3.35 (1) and 3.25 (1) Å; C(5)···O(2)  $(x, y, -1+z)$  3.39 (1) Å, and C(3)···O(3),

C(3a)···O(3),  $(1+x, 1.5-y, 0.5+z)$  3.13 (1) and 3.26 (1) Å. No suitable H donors for other H-bond formation are available. The molecules are held in the crystal by van der Waals forces.

Financial support from the Consejo Nacional de Ciencia y Tecnología de México, CONACYT (Project No. PCEXECNA-040603) is acknowledged. We thank Messrs R. A. Toscano and Abelardo Cuellar for technical assistance.

#### References

- ALTONA, C., GEISE, H. J. & ROMERS, C. (1968). *Tetrahedron*, **24**, 13–32.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- SHELDRICK, G. M. (1985). *SHELXTL5. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data*, revision 5. Univ. of Göttingen, Federal Republic of Germany.
- SUTTON, L. E. (1965). *Tables of Interatomic Distances and Configuration in Molecules and Ions*. Special Publication No. 18. London: The Chemical Society.
- YUSTE, F. & SÁNCHEZ-OBREGÓN, R. (1982). *J. Org. Chem.* **47**, 3665–3668.

*Acta Cryst.* (1989). **C45**, 900–902

## Structure of 5-(3,4-Methylenedioxyphenyl)-1-piperidinopent-2,4-dien-1-one

BY M. NETHAJI AND VASANTHA PATTABHI\*

*Department of Crystallography and Biophysics,† University of Madras, Guindy Campus, Madras-600 025, India*

(Received 20 September 1988; accepted 6 December 1988)

**Abstract.** C<sub>17</sub>H<sub>19</sub>NO<sub>3</sub>,  $M_r = 285.35$ , monoclinic,  $P2_1/n$ ,  $a = 8.740$  (1),  $b = 13.647$  (1),  $c = 13.163$  (1) Å,  $\beta = 108.77$  (1)°,  $V = 1486.6$  (3) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.275 \text{ g cm}^{-3}$ ,  $\text{Cu K}\alpha$ ,  $\lambda = 1.5418 \text{ \AA}$ ,  $\mu = 6.7 \text{ cm}^{-1}$ ,  $F(000) = 608$ ,  $T = 295 \text{ K}$ ,  $R(F) = 0.041$ ,  $wR = 0.044$  for 996 unique reflections with  $I \geq 2.5\sigma(I)$ . The piperidine ring is in a chair conformation, the 3,4-methylenedioxyphenyl group is nearly planar, and the dihedral angle between these is  $45.3$  (1)°. The crystal structure is stabilized by van der Waals forces only.

**Introduction.** The title compound, which is a natural product, is extracted from the plant *Vicou Indica* Dc. The present study was undertaken to confirm the structure proposed by chemists (Purushothaman, 1988).

**Experimental.** Pale-green needles  $0.17 \times 0.13 \times 0.1 \text{ mm}$  from methanol, three-dimensional intensity data on Enraf-Nonius CAD-4 automatic diffractometer,  $\omega/2\theta$  scan mode with  $2 \leq 2\theta \leq 120^\circ$ ,  $\text{Cu K}\alpha$  radiation, data corrected for direct-beam polarization and Lorentz effects, and for absorption (average transmission factor 0.926, with maximum and minimum 0.861 and 0.999, respectively),  $0 \leq h \leq 9$ ,  $0 \leq k \leq 14$ ,  $-13 \leq l \leq 13$ , 1753 measured reflections, 1471 unique and 996 observed with  $I \geq 2.5\sigma(I)$ , cell constants from 16 reflections with  $28 \leq 2\theta \leq 59^\circ$ , three standard reflections monitored for every 100 reflections showed no significant variation in intensity, scan width using the relation  $(A + B \tan \theta)$  where  $A$  and  $B$  are 0.8 and 0.14, respectively, aperture width using  $(4.0 + 2 \tan \theta)$ , maximum time spent on any reflection measurement was 30 s, and the background count was half the scan time. Structure solution by direct methods, H atoms from  $\Delta\rho$  synthesis, full-matrix least-squares

\* To whom correspondence should be addressed.

† DCB contribution No. 725.