

1,50, $r(O \leq) = 1,52$ et $r(F) = 1,47$ Å, et si, parmi les sites fluorés, on ne retient que ceux dont le taux d'occupation est le plus élevé, on observe que cinq distances interatomiques intermoléculaires seulement diffèrent de moins de 0,1 Å de la somme des rayons des atomes concernés. Il s'agit de C(2)...C(3ⁱ) [3,546 (3) Å] [(i): 1-x, 1-y, 1-z], de C(6)...O(24ⁱ) [3,341 (3) Å], de C(8)...C(8ⁱⁱ) [3,474 (2) Å] [(ii): 1-x, -y, 1-z], de C(11)...C(14ⁱⁱ) [3,515 (3) Å] et de C(12)...O(24ⁱⁱ) [3,287 (3) Å].

Références

- ARGAY, GY. & SERES, J. (1973). *Acta Cryst.* B29, 1146-1149.
 BONDI, A. (1964). *J. Phys. Chem.* pp. 441-451.
 BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1964). *ORFFE*. Rapport ORNL-TM-306. Oak Ridge National Laboratory, Tennessee, EU.
 DHANARAJ, V. & VIJAYAN, M. (1983). *Acta Cryst.* C39, 1398-1401.
 FRENZ, B. A. (1982). *SDP Structure Determination Package*. B. A. Frenz & Associates, College Station, Texas, EU, et Enraf-Nonius, Delft, Pays-Bas.

- International Tables for X-ray Crystallography* (1974). Tome IV, pp. 99 et 149. Birmingham: Kynoch Press. (Distributeur actuel D. Reidel, Dordrecht.)
 ITO, T. & SUGAWARA, Y. (1983). *Best-Plane Program*, 3^e version (BP7C). The Institute of Physical and Chemical Research, Wako-Shi, Saitama 351, Japon.
 JOHNSON, C. K. (1976). *ORTEPII*. Rapport ORNL-5138. Oak Ridge National Laboratory, Tennessee, EU.
 KRISHNA MURTHY, H. M., BHAT, T. N. & VIJAYAN, M. (1982). *Acta Cryst.* B38, 315-317.
 KRISHNA MURTHY, H. M. & VIJAYAN, M. (1979). *Acta Cryst.* B35, 262-263.
 KRISHNA MURTHY, H. M. & VIJAYAN, M. (1981). *Acta Cryst.* B37, 1102-1105.
 MCCONNELL, J. F. (1973). *Cryst. Struct. Commun.* 2, 459-461.
 MCCONNELL, J. F. & COMPANY, F. Z. (1976). *Cryst. Struct. Commun.* 5, 861-864.
 MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCO, J.-P. & WOOLFSON, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univ. de York, Angleterre, et de Louvain, Belgique.
 MICHEL, A., GUSTIN, R., EVRARD, G. & DURANT, F. (1982). *Bull. Soc. Chim. Belg.* 91, 123-129.
 MORSSINK, H. & VAN BODEGOM, B. (1981). *Acta Cryst.* B37, 107-114.

Acta Cryst. (1988). C44, 550-553

Structures of 2-Ethyl-4,5-dihydro-5-phenyl-3H-1,4-benzodiazepin-3-one (I) and 2-Ethyl-1,4,5-trihydro-2,5-diphenyl-3H-1,4-benzodiazepin-3-one (II)

BY KARL WILHELM TÖRNROOS

Department of Structural Chemistry, Arrhenius Laboratory, University of Stockholm, S-106 91 Stockholm, Sweden

(Received 3 August 1987; accepted 16 November 1987)

Abstract. (I): C₁₇H₁₆N₂O, $M_r = 264.3$, monoclinic, $P2_1/c$, $a = 10.306$ (1), $b = 8.781$ (1), $c = 15.677$ (3) Å, $\beta = 90.41$ (2)°, $V = 1418.7$ (3) Å³, $Z = 4$, $D_x = 1.237$ g cm⁻³, $\lambda(\text{Cu K}\alpha) = 1.54184$ Å, $\mu = 5.83$ cm⁻¹, $F(000) = 560$, $R = 0.0545$, $wR = 0.0547$ for 2090 reflections. (II): C₂₃H₂₂N₂O, $M_r = 342.4$, triclinic, $P\bar{1}$, $a = 12.979$ (1), $b = 8.399$ (1), $c = 9.517$ (1) Å, $\alpha = 111.04$ (1), $\beta = 90.21$ (1), $\gamma = 105.82$ (1)°, $V = 925.6$ (2) Å³, $Z = 2$, $D_x = 1.229$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.71069$ Å, $\mu = 0.705$ cm⁻¹, $F(000) = 364$, $R = 0.0480$ for 3163 reflections. Probably because of different hybridization of atoms N1 and C2 in the two compounds the diazepine ring in compound (I) adopts a boat conformation, and an envelope conformation in compound (II). The double bond between N1 and C2 in (I), not present in (II), conjugates with the carbonyl bond C3-O12, and results in a shortening of bonds C2-C3, C2-C10 as well as C10-C11 compared with (II). Both structures dimerize by means of two hydrogen bonds between the two enantiomers.

Introduction. The pharmacological properties of 1,4-benzodiazepin-2-ones have prompted a large number of investigations, pharmacological (Priest, Vianna Filho, Amrein & Skreta, 1979) as well as structural (Gałdecki & Głowka, 1980). This paper is based on a study of 1,4-benzodiazepin-3-ones, on the routes of chemical syntheses and conformational effects on the benzodiazepine ring system, as a function of varying substituent schemes. The chemical syntheses of the title compounds have been described by Bergman, Brynolf & Elman (1983).

Experimental. (I): Colourless prisms from CH₃CN, crystal specimen size 0.50 × 0.34 × 0.12 mm. Philips PW1100 four-circle diffractometer, graphite-monochromatized Cu Kα radiation. Cell parameters from least-squares refinement of 23 diffractometer-centred reflections ($12.105 \leq \theta \leq 32.605^\circ$). $\omega/2\theta$ scan, scan width 1.60°, scan speed 1.8° min⁻¹. 2885 reflections measured, 2436 unique, $5.04 \leq \theta \leq 66.97^\circ$, $0 \leq h \leq$

12, $0 \leq k \leq 10$, $-18 \leq l \leq 18$. Three standard reflections monitored every 90 min, no significant intensity variation. No absorption correction. 2090 reflections with $|F| \geq 6\sigma(F)$ accepted as significant. (II): Colourless prisms from CH_3CN , crystal specimen size $0.75 \times 0.50 \times 0.38$ mm. $\text{Mo K}\alpha$ radiation. Cell parameters by least-squares refinement of 20 reflections centered on diffractometer ($13.04 \leq \theta \leq 24.50^\circ$). Data collection by $\omega/2\theta$ scan, scan width 1.00° , scan speed $1.2^\circ \text{ min}^{-1}$. 5631 reflections measured, $2.72 \leq \theta \leq 30.00^\circ$, $0 \leq h \leq 17$, $-11 \leq k \leq 10$, $-13 \leq l \leq 13$, three standard reflections monitored every 60 min, maximum intensity variation $\pm 4\%$. No absorption correction. 3163 unique reflections with $|F| \geq 6\sigma(F)$ considered observed.

Both structures solved by *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Full-matrix least-squares refinement, minimizing $\sum w(\Delta F)^2$, with *SHELX76* (Sheldrick, 1976),

Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors with e.s.d.'s in parentheses

$$B_{\text{eq}} = \frac{4}{3} \sum_i \sum_j \beta_{ij} a_i a_j$$

Compound (I)	x	y	z	$B_{\text{eq}}(\text{\AA}^2)$
N1	0.8868 (1)	0.1311 (2)	0.1070 (1)	3.43 (4)
C2	0.8207 (2)	0.1369 (2)	0.0380 (1)	3.47 (5)
C3	0.6851 (2)	0.0733 (2)	0.0245 (1)	3.72 (5)
N4	0.5946 (1)	0.1033 (2)	0.0818 (1)	3.73 (5)
C5	0.6159 (2)	0.1764 (2)	0.1644 (1)	3.30 (4)
C5a	0.7160 (2)	0.0877 (2)	0.2155 (1)	3.29 (5)
C6	0.6839 (2)	0.0233 (2)	0.2935 (1)	4.23 (6)
C7	0.7737 (3)	-0.0601 (3)	0.3396 (1)	5.16 (7)
C8	0.8984 (3)	-0.0784 (3)	0.3090 (1)	5.28 (7)
C9	0.9320 (2)	-0.0124 (3)	0.2327 (1)	4.44 (6)
C9a	0.8415 (2)	0.0684 (2)	0.1843 (1)	3.38 (5)
C10	0.8798 (2)	0.1975 (3)	-0.0422 (1)	4.77 (6)
C11	1.0126 (2)	0.2657 (3)	-0.0328 (2)	5.37 (7)
O12	0.6629 (1)	-0.0005 (2)	-0.0413 (1)	5.36 (5)
C1''	0.6455 (2)	0.3465 (2)	0.1595 (1)	3.34 (4)
C2''	0.6283 (2)	0.4322 (3)	0.0862 (1)	4.48 (6)
C3''	0.6498 (3)	0.5895 (3)	0.0879 (2)	5.47 (7)
C4''	0.6881 (2)	0.6599 (3)	0.1629 (2)	5.59 (7)
C5''	0.7039 (2)	0.5760 (3)	0.2357 (2)	5.07 (6)
C6''	0.6832 (2)	0.4203 (2)	0.2344 (1)	4.28 (6)

Compound (II)	x	y	z	$B_{\text{eq}}(\text{\AA}^2)$
N1	0.3282 (1)	0.3215 (2)	0.4417 (2)	3.11 (5)
C2	0.2390 (2)	0.1749 (3)	0.3377 (2)	2.96 (6)
C3	0.1424 (2)	0.1072 (3)	0.4177 (2)	3.09 (5)
N4	0.1335 (1)	0.1874 (2)	0.5643 (3)	3.15 (5)
C5	0.2096 (2)	0.3362 (3)	0.6876 (2)	3.06 (6)
C5a	0.3204 (2)	0.3125 (3)	0.6913 (2)	3.14 (6)
C6	0.3687 (2)	0.3108 (3)	0.8216 (3)	4.11 (7)
C7	0.4734 (2)	0.3005 (3)	0.8274 (3)	4.96 (9)
C8	0.5299 (2)	0.2916 (3)	0.7035 (3)	5.1 (1)
C9	0.4825 (2)	0.2915 (3)	0.5717 (3)	4.27 (8)
C9a	0.3771 (2)	0.3024 (3)	0.5656 (2)	3.16 (6)
C10	0.2781 (2)	0.0118 (3)	0.2479 (3)	4.14 (7)
C11	0.3695 (2)	0.0523 (4)	0.1524 (3)	5.6 (1)
O12	0.0699 (1)	-0.0265 (2)	0.3376 (2)	4.39 (5)
C1''	0.2071 (2)	0.5187 (3)	0.6915 (2)	3.18 (6)
C2''	0.1092 (2)	0.5454 (3)	0.6676 (3)	4.67 (8)
C3''	0.1037 (2)	0.7099 (4)	0.6718 (4)	5.8 (1)
C4''	0.1968 (2)	0.8485 (3)	0.6986 (3)	5.5 (1)
C5''	0.2950 (2)	0.8244 (3)	0.7248 (3)	5.2 (1)
C6''	0.3003 (2)	0.6609 (3)	0.7237 (3)	4.05 (7)
C21	0.1931 (2)	0.2514 (3)	0.2349 (2)	3.12 (6)
C22	0.1410 (2)	0.1416 (3)	0.0904 (2)	3.95 (6)
C23	0.0957 (2)	0.2108 (4)	0.0019 (3)	4.78 (9)
C24	0.1028 (2)	0.3902 (4)	0.0561 (3)	5.2 (1)
C25	0.1535 (2)	0.5005 (4)	0.1996 (3)	5.0 (1)
C26	0.1983 (2)	0.4315 (3)	0.2896 (3)	3.85 (7)

with non-hydrogen atoms anisotropic, H atoms in calculated positions with isotropic group temperature factors, except for H4 and H5 [structure(I)] and H1, H4 and H5 [structure(II)], which were taken from $\Delta\rho$ map and individually and isotropically refined. Structure (I): $R = 0.0545$, $wR = 0.0457$, $w = 1/[\sigma^2(F) + 0.0003F^2]$, for 221 parameters. Max. Δ/σ for non-

Table 2. Bond lengths (\AA), angles ($^\circ$) and hydrogen-bond data with e.s.d.'s in parentheses

Compound (I)			
N1-C2	1.275 (2)	C6-C7	1.380 (4)
N1-C9a	1.414 (2)	C7-C8	1.385 (4)
C2-C10	1.499 (3)	C8-C9	1.375 (3)
C2-C3	1.518 (3)	C9-C9a	1.394 (3)
C3-O12	1.238 (3)	C10-C11	1.501 (3)
C3-N4	1.326 (2)	C1''-C2''	1.383 (3)
N4-C5	1.460 (2)	C1''-C6''	1.394 (3)
C5-C5a	1.517 (3)	C2''-C3''	1.400 (4)
C5-C1''	1.526 (3)	C3''-C4''	1.384 (4)
C5a-C6	1.389 (3)	C4''-C5''	1.367 (4)
C5a-C9a	1.396 (3)	C5''-C6''	1.384 (3)
C2-N1-C9a	124.4 (2)	C5a-C6-C7	121.0 (2)
N1-C2-C3	126.2 (2)	C6-C7-C8	120.0 (2)
N1-C2-C10	120.5 (2)	C7-C8-C9	119.6 (2)
C10-C2-C3	113.1 (2)	C8-C9-C9a	121.1 (2)
C2-C3-N4	118.9 (2)	N1-C9a-C5a	124.4 (2)
C2-C3-O12	118.3 (2)	N1-C9a-C9	116.2 (2)
O12-C3-N4	122.8 (2)	C5a-C9a-C9	119.3 (2)
C3-N4-C5	125.9 (2)	C2-C10-C11	115.8 (2)
N4-C5-C1''	114.5 (2)	C2''-C1''-C6''	118.7 (2)
N4-C5-C5a	109.8 (2)	C1''-C2''-C3''	120.2 (2)
C5a-C5-C1''	113.2 (2)	C2''-C3''-C4''	120.0 (2)
C5-C5a-C9a	120.4 (2)	C3''-C4''-C5''	120.0 (2)
C5-C5a-C6	120.6 (2)	C4''-C5''-C6''	120.2 (2)
C6-C5a-C9a	119.0 (2)	C1''-C6''-C5''	120.9 (2)

Compound (II)			
N1-C2	1.474 (2)	C9-C9a	1.398 (3)
N1-C9a	1.417 (3)	C10-C11	1.537 (4)
C2-C3	1.550 (3)	C1''-C2''	1.381 (3)
C2-C10	1.551 (3)	C1''-C6''	1.389 (3)
C2-C21	1.543 (3)	C2''-C3''	1.389 (4)
C3-N4	1.336 (3)	C3''-C4''	1.378 (3)
C3-O12	1.238 (2)	C4''-C5''	1.379 (4)
N4-C5	1.476 (2)	C5''-C6''	1.390 (4)
C5-C1''	1.529 (3)	C21-C22	1.394 (3)
C5-C5a	1.508 (3)	C21-C26	1.394 (3)
C5a-C9a	1.396 (3)	C22-C23	1.388 (4)
C5a-C6	1.393 (3)	C23-C24	1.383 (4)
C6-C7	1.387 (4)	C24-C25	1.382 (3)
C7-C8	1.382 (4)	C25-C26	1.394 (4)
C8-C9	1.395 (4)		
C2-N1-C9a	120.8 (2)	C7-C8-C9	120.7 (2)
N1-C2-C3	114.0 (2)	C8-C9-C9a	119.6 (2)
N1-C2-C10	111.0 (2)	N1-C9a-C5a	117.6 (2)
N1-C2-C21	106.8 (2)	N1-C9a-C9	122.5 (2)
C3-C2-C10	107.0 (2)	C5a-C9a-C9	119.6 (2)
C3-C2-C21	104.8 (2)	C2-C10-C11	114.1 (2)
C10-C2-C21	113.2 (2)	C2''-C1''-C6''	118.8 (2)
C2-C3-N4	123.6 (2)	C1''-C2''-C3''	120.8 (2)
C2-C3-O12	116.6 (2)	C2''-C3''-C4''	120.0 (3)
N4-C3-O12	119.9 (2)	C3''-C4''-C5''	119.7 (3)
C3-N4-C5	131.6 (2)	C4''-C5''-C6''	120.2 (2)
N4-C5-C5a	112.1 (2)	C1''-C6''-C5''	120.3 (2)
C1''-C5-N4	112.5 (2)	C22-C21-C26	119.0 (2)
C1''-C5-C5a	114.9 (2)	C21-C22-C23	120.5 (2)
C5-C5a-C6	120.7 (2)	C22-C23-C24	120.1 (2)
C5-C5a-C9a	119.3 (2)	C23-C24-C25	120.0 (3)
C6-C5a-C9a	120.0 (2)	C24-C25-C26	120.2 (3)
C5a-C6-C7	120.3 (2)	C21-C26-C25	120.2 (2)
C6-C7-C8	119.8 (2)		

	Compound (I)	Compound (II)
N4-H4...O12 (\AA)	2.870 (2)	2.940 (2)
N4-H4 (\AA)	0.82 (2)	0.90 (2)
H4...O12 (\AA)	2.05 (2)	2.04 (2)
N4-H4...O12 ($^\circ$)	173 (2)	179 (2)
Symmetry operator of the second molecule	$-x + 1, -y, -z$	$-x, -y, -z + 1$

hydrogen atoms 0.001, residual electron density in final $\Delta\rho$ map 0.20 and -0.29 e \AA^{-3} . Structure (II): $R = 0.0480$, unit weights, refining 286 parameters. Max. Δ/σ for non-hydrogen atoms 0.001, max. and min. peaks in final $\Delta\rho$ map $\pm 0.20 \text{ e \AA}^{-3}$.

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). All geometrical calculations made with *PARST* (Nardelli, 1983), all figures drawn with *PLUTO* (Motherwell & Clegg, 1978).

Discussion. Final atomic coordinates and thermal parameters for non-hydrogen atoms are given in Table 1.* Table 2 lists bond lengths, angles and hydrogen-

* 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 44541 (97 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

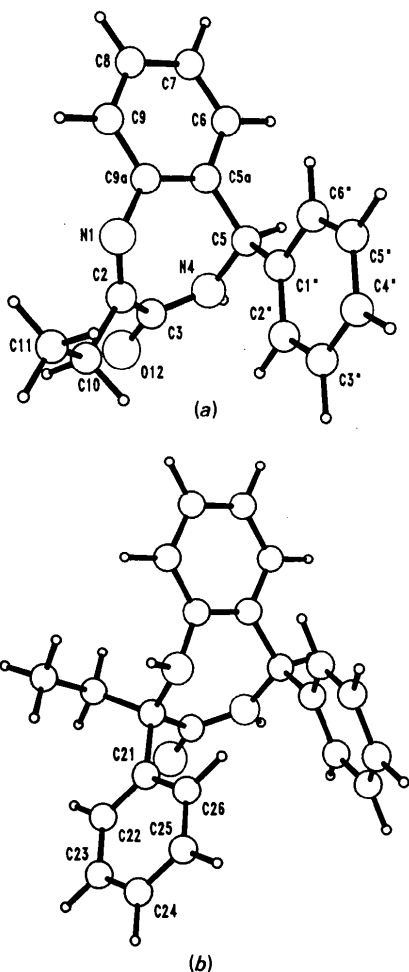


Fig. 1. Perspective view: (a) compound (I); (b) compound (II). Numbering scheme of (II) is identical to (I) except for indicated atoms.

bond data. Views of the compounds with labelling of the atoms are shown in Fig. 1. Fig. 2 illustrates the dimerization of the enantiomers through hydrogen bonds. The two compounds are similarly substituted, except for the addition of a phenyl group at position C2 in (II). A second difference is the subsequent absence of a double bond between N1—C2 in (II). This difference in hybridization is probably the contributory cause for the different conformations of the diazepine ring. Compound (I) has a boat-shaped diazepine ring and compound (II) has an envelope conformation.

A comparison of torsion angles reveals that the angle C5a—C9a—N1—C2 differs substantially, $40.4 (3)^\circ$ (I) and $75.5 (2)^\circ$ (II). The opposite angle, C9a—C5a—C5—N4, differs by only $0.3 (2)^\circ$. Owing to the double bond between N1 and C2 in (I), which conjugates with the carbonyl bond C3—O12, the distance C2—C3 is $0.032 (3) \text{ \AA}$ shorter than in (II). Furthermore, this conjugation seems to affect the bond lengths of the ethyl group, giving a shortening of C2—C10 by $0.052 (3) \text{ \AA}$ and C10—C11 by $0.036 (3) \text{ \AA}$ compared with (II).

In (II) the additional and more bulky phenyl group at C2 takes the same position as the ethyl group in (I), thus pushing the ethyl group to a *trans* position in relation to (I). The indication that the envelope conformation in (II) is due to the hybridization of N1 and C2 can also be seen in conformer *B* of an earlier studied 1,4-benzodiazepin-3-one (Bergman, Brynolf, Törnroos, Karlsson & Werner, 1983), which is hybridized in the same way.

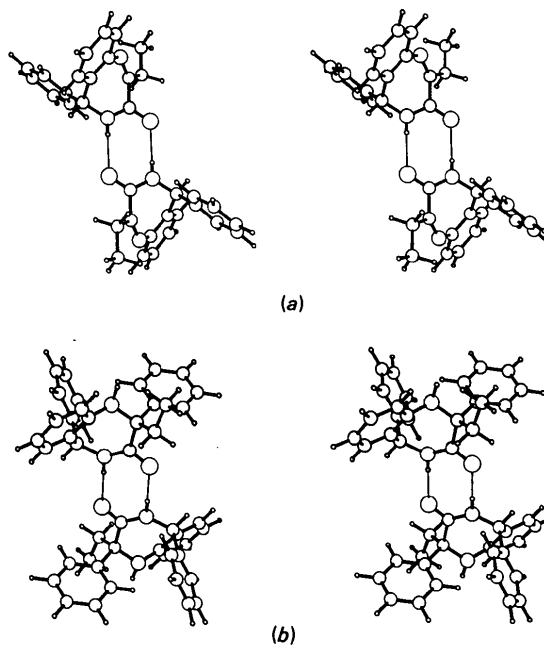


Fig. 2. Stereoscopic view of the hydrogen-bonded dimers: (a) compound (I); (b) compound (II).

The benzene ring of compound (I) deviates statistically significantly from planarity with a maximum departure from the plane of 0.015 (2) Å (C9). The benzene ring in compound (II) is planar. The molecules in both structures are dimerized through a pair of hydrogen bonds with the opposite enantiomer; see Table 2 for bond data.

References

- BERGMAN, J., BRYNOLF, A. & ELMAN, B. (1983). *Heterocycles*, **20**, 2141–2144.
- BERGMAN, J., BRYNOLF, A., TÖRNROOS, K. W., KARLSSON, B. & WERNER, P.-E. (1983). *Heterocycles*, **20**, 2145–2148.
- GALDECKI, Z. & GLÓWKA, M. L. (1980). *Acta Cryst.* **B36**, 3044–3048.
- International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- NARDELLI, M. (1983). *Comput. Chem.* **7**, 95–98.
- PRIEST, R. G., VIANNA FILHO, U., AMREIN, R. & SKRETA, M. (1979). *Benzodiazepines Today and Tomorrow*. Proc. 1st Int. Symp. on Benzodiazepines, Rio de Janeiro 28–30 Sept. 1979. MTP Press Limited.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1988). **C44**, 553–555

(+)-Jaborosalactone M, a Hemiketal Withanolide from *Jaborosa magellanica*

By MASOOD PARVEZ, VICTOR FAJARDO AND MAURICE SHAMMA

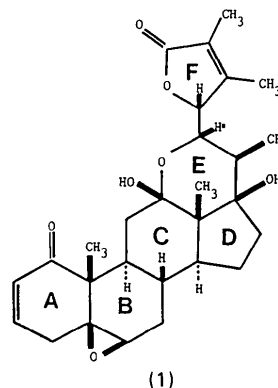
Department of Chemistry, The Pennsylvania State University, University Park, PA 16802, USA

(Received 27 July 1987; accepted 18 November 1987)

Abstract. $C_{28}H_{36}O_7$, $M_r = 484.59$, orthorhombic, $P2_12_12_1$, $a = 11.444$ (4), $b = 11.920$ (4), $c = 18.556$ (6) Å, $V = 2531$ (3) Å³, $Z = 4$, $D_m = 1.26$, $D_x = 1.27$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.084$ mm⁻¹, $F(000) = 1040$, $T = 293$ (1) K, $R = 0.037$ for 2201 observed reflections with $I > 3\sigma(I)$. The crystal structure consists of discrete molecules of (+)-jaborosalactone M separated by normal van der Waals distances. The mean bond lengths are C(sp³)–C(sp³) 1.532 (3), C(sp³)–C(sp²) 1.501 (3), C(sp²)–C(sp²) 1.464 (4), C=C 1.312 (4), C(sp³)–O 1.448 (3), C(sp²)–O 1.360 (3) and C=O 1.211 (3) Å. Ring A is in a twist-boat conformation, ring B is a half-chair, ring C a regular chair, and ring D is a C(13)-envelope. The hemiketal ring E is a chair while the lactonic ring F is essentially planar. Atoms C(18), C(19), O(3) and O(4) are β and O(5) and the lactone moiety are α oriented. Both the hydroxy groups are involved in hydrogen bonding [O(1)⋯H(O3ⁱ) 2.18 and O(3ⁱ)⋯H(O4) 2.10 Å].

Introduction. The withanolides are C₂₈ steroids found among members of the Solanaceae (Tursunova, Maslennikova & Abubakirov, 1977; Kamernitskii, Reshetova & Krivoruchko, 1977; Vasina, Maslennikova & Abubakirov, 1986). An investigation of *Jaborosa magellanica* (Griseb.) Dusen (Solanaceae), collected along the Strait of Magellan, Chile, has

yielded a wide variety of new withanolides. We now describe the first of these novel withanolides, (+)-jaborosalactone M, whose most unusual feature is the presence of a six-membered hemiketal ring E. We did not establish the absolute configuration by anomalous scattering. Rather, the configuration indicated in (1) derives from the fact that naturally occurring steroidal derivatives generally incorporate a C(18) methyl group which lies above the mean plane of the molecule.



Experimental. Colorless prismatic crystals of (+)-jaborosalactone M for X-ray crystallographic study were grown by slow evaporation of an ethanol:ethyl acetate