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

O(6)–C(5*a*)	1·343 (2)	O(6)–C(7)	1·456 (2)
C(5*a*)–O(5)	1·336 (2)	C(5*a*)–C(10*b*)	1·355 (2)
O(5)–C(4)	1·424 (2)	N(2)–C(1)	1·352 (2)
N(2)–C(2*n*)	1·453 (2)	N(2)–C(3)	1·460 (2)
O(1)–C(1)	1·234 (2)	C(1)–C(10*b*)	1·491 (2)
C(10*b*)–C(10*a*)	1·532 (2)	C(4)–C(1*p*)	1·515 (2)
C(4)–C(3)	1·516 (2)	C(1*p*)–C(2*p*)	1·378 (3)
C(1*p*)–C(6*p*)	1·379 (3)	C(10*a*)–C(7*a*)	1·541 (3)
C(10*a*)–C(10)	1·542 (3)	C(7)–C(7″)	1·513 (3)
C(7)–C(7*a*)	1·515 (3)	C(7)–C(7′)	1·509 (4)
C(3)–C(3′)	1·526 (3)	C(2*p*)–C(3*p*)	1·379 (3)
C(6*p*)–C(5*p*)	1·400 (3)	C(7*a*)–C(8)	1·530 (4)
C(3*p*)–C(4*p*)	1·374 (3)	C(5*p*)–C(4*p*)	1·359 (3)
C(10)–C(9*a*)	1·477 (5)	C(8)–C(9*a*)	1·491 (6)
C(7)–O(6)–C(5*a*)	117·7 (1)	O(5)–C(5*a*)–O(6)	105·2 (1)
C(10*b*)–C(5*a*)–O(6)	124·2 (2)	C(10*b*)–C(5*a*)–O(5)	130·6 (2)
C(4)–O(5)–C(5*a*)	118·8 (1)	C(2*n*)–N(2)–C(1)	117·3 (1)
C(3)–N(2)–C(1)	126·4 (1)	C(3)–N(2)–C(2*n*)	115·7 (1)
O(1)–C(1)–N(2)	119·0 (2)	C(10*b*)–C(1)–N(2)	123·8 (1)
C(10*b*)–C(1)–O(1)	117·1 (2)	C(1)–C(10*b*)–C(5*a*)	129·6 (1)
C(10*a*)–C(10*b*)–C(5*a*)	118·4 (2)	C(10*a*)–C(10*b*)–C(1)	111·9 (1)
C(1*p*)–C(4)–O(5)	107·9 (1)	C(3)–C(4)–O(5)	111·6 (2)
C(3)–C(4)–C(1*p*)	111·0 (1)	C(2*p*)–C(1*p*)–C(4)	118·5 (2)
C(6*p*)–C(1*p*)–C(4)	122·1 (2)	C(6*p*)–C(1*p*)–C(2*p*)	119·2 (2)
C(7*a*)–C(10*a*)–C(10*b*)	113·5 (1)	C(10)–C(10*a*)–C(10*b*)	112·7 (2)
C(10)–C(10*a*)–C(7*a*)	103·9 (2)	C(7″)–C(7)–O(6)	103·9 (2)
C(7*a*)–C(7)–O(6)	107·2 (2)	C(7*a*)–C(7)–C(7″)	113·6 (2)
C(7′)–C(7)–O(6)	108·9 (2)	C(7′)–C(7)–C(7″)	110·5 (2)
C(7′)–C(7)–C(7*a*)	112·2 (2)	C(4)–C(3)–N(2)	112·3 (1)
C(3′)–C(3)–N(2)	111·8 (2)	C(3′)–C(3)–C(4)	113·1 (2)
C(3*p*)–C(2*p*)–C(1*p*)	120·6 (2)	C(5*p*)–C(6*p*)–C(1*p*)	119·5 (2)
C(7)–C(7*a*)–C(10*a*)	112·8 (2)	C(8)–C(7*a*)–C(10*a*)	104·5 (2)
C(8)–C(7*a*)–C(7)	116·1 (2)	C(4*p*)–C(3*p*)–C(2*p*)	120·3 (2)
C(4*p*)–C(5*p*)–C(6*p*)	120·7 (2)	C(5*p*)–C(4*p*)–C(3*p*)	119·6 (2)
C(9*a*)–C(10)–C(10*a*)	107·3 (2)	C(9*a*)–C(8)–C(7*a*)	102·3 (3)
C(8)–C(9*a*)–C(10)	106·6 (3)		

Related literature. For the preparation of the compound see Pfeiffer (1988). For the preparation of some related compounds see Tietze, Brand, Pfeiffer, Antel, Harms & Sheldrick (1987).

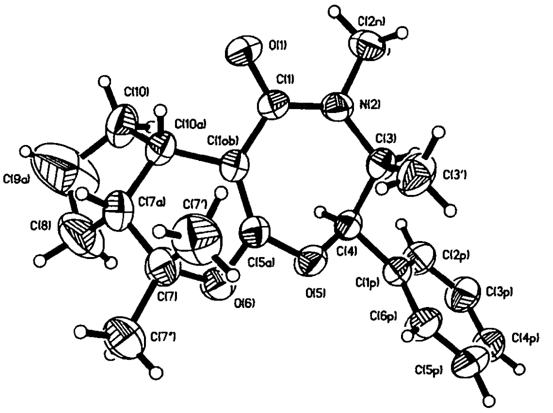

Fig. 1. The molecular structure showing atom-numbering scheme.

We thank the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie for financial support.

References

Clegg, W. (1981). *Acta Cryst.* A**37**, 22–28.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Pfeiffer, T. (1988). PhD thesis. Univ. of Göttingen, Federal Republic of Germany.

Sheldrick, G. M. (1976). *SHELX76*. Crystal structure refinement program modified by GMS. Univ. of Cambridge, England.

Sheldrick, G. M. (1985). *SHELXS86*. In *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford Univ. Press.

Tietze, L.-F., Brand, S., Pfeiffer, T., Antel, J., Harms, K. & Sheldrick, G. M. (1987). *J. Am. Chem. Soc.* **109**, 921.

Acta Cryst. (1988). C**44**, 2227–2229

Structure of a Furo[3,4-*b*]azepine Derivative

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(*Received* 26 *April* 1988; *accepted* 15 *July* 1988)

Abstract. (5a*RS*,8a*RS*)-Ethyl 6,6-dimethyl-8-oxo-perhydrofuro[3,4-*b*]azepine-8a-carboxylate, $C_{13}H_{21}NO_4$, $M_r = 255\cdot16$, monoclinic, $P2_1/c$, $a = 7\cdot189$ (1), $b = 15\cdot057$ (2), $c = 13\cdot101$ (2) Å, $\beta = 96\cdot62$ (3)°, $V = 1408\cdot65$ Å^3, $Z = 4$, $D_x = 1\cdot204$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0\cdot71069$ Å, $\mu = 0\cdot08$ mm^{-1}, $F(000) = 552$, $T =$

0108-2701/88/122227-03$03.00

Table 1. *Atomic coordinates* (×10^4) *and equivalent isotropic displacement parameters* (Å^2 × 10^3)

	x	y	z	U_{eq}*
N(1)	−463 (4)	−56 (1)	1772 (2)	63 (1)
C(2)	207 (5)	−806 (2)	2435 (3)	85 (1)
C(3)	1767 (6)	−1303 (2)	2010 (3)	95 (1)
C(4)	3487 (5)	−774 (2)	1906 (3)	90 (1)
C(5)	3199 (4)	38 (2)	1208 (2)	70 (1)
C(5a)	2479 (3)	859 (2)	1729 (2)	51 (1)
C(6)	2498 (3)	1731 (2)	1100 (2)	55 (1)
C(6′)	3549 (4)	2472 (2)	1714 (3)	82 (1)
C(6″)	3157 (4)	1658 (2)	47 (2)	73 (1)
O(7)	532 (2)	2016 (1)	910 (2)	76 (1)
C(8)	−617 (3)	1476 (2)	1327 (2)	57 (1)
O(8′)	−2280 (2)	1545 (2)	1167 (2)	82 (1)
C(8a)	469 (3)	784 (2)	2008 (2)	49 (1)
C(9)	363 (4)	1080 (2)	3136 (2)	66 (1)
O(10)	1627 (3)	1368 (2)	3696 (2)	116 (1)
O(11)	−1310 (3)	972 (2)	3403 (2)	92 (1)
C(12)	−1622 (6)	1231 (3)	4439 (3)	110 (2)
C(13)	−3409 (7)	1076 (5)	4610 (4)	186 (4)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

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

N(1)—C(2)	1·472 (4)	N(1)—C(8a)	1·448 (3)
C(2)—C(3)	1·507 (5)	C(3)—C(4)	1·490 (5)
C(4)—C(5)	1·526 (4)	C(5)—C(5a)	1·531 (4)
C(5a)—C(6)	1·551 (3)	C(5a)—C(8a)	1·535 (3)
C(6)—C(6′)	1·524 (4)	C(6)—C(6″)	1·513 (4)
C(6)—O(7)	1·471 (3)	O(7)—O(8)	1·321 (3)
C(8)—O(8′)	1·194 (3)	C(8)—C(8a)	1·527 (3)
C(8a)—C(9)	1·553 (4)	C(9)—O(10)	1·182 (4)
C(9)—O(11)	1·302 (4)	O(11)—C(12)	1·453 (4)
C(12)—C(13)	1·350 (6)		
		C(2)—N(1)—C(8a)	115·6 (2)
N(1)—C(2)—C(3)	111·7 (3)	C(2)—C(3)—C(4)	115·5 (3)
C(3)—C(4)—C(5)	115·2 (3)	C(4)—C(5)—C(5a)	114·2 (3)
C(5)—C(5a)—C(6)	115·0 (2)	C(5)—C(5a)—C(8a)	115·0 (2)
C(6)—C(5a)—C(8a)	104·8 (2)	C(5a)—C(6)—C(6′)	111·9 (2)
C(5a)—C(6)—C(6″)	116·5 (2)	C(6′)—C(6)—C(6″)	110·2 (2)
C(5a)—C(6)—O(7)	105·6 (2)	C(6′)—C(6)—O(7)	106·5 (2)
C(6″)—C(6)—O(7)	105·2 (2)	C(6)—O(7)—C(8)	112·7 (2)
O(7)—C(8)—O(8′)	122·4 (2)	O(7)—C(8)—C(8a)	111·1 (2)
O(8′)—C(8)—C(8a)	126·6 (2)	N(1)—C(8a)—C(5a)	116·0 (2)
N(1)—C(8a)—C(8)	106·2 (2)	C(5a)—C(8a)—C(8)	104·0 (2)
N(1)—C(8a)—C(9)	112·5 (2)	C(5a)—C(8a)—C(9)	110·8 (2)
C(8)—C(8a)—C(9)	106·3 (2)	C(8a)—C(9)—O(10)	125·3 (3)
C(8a)—C(9)—O(11)	111·6 (2)	O(10)—C(9)—O(11)	123·1 (3)
C(9)—O(11)—C(12)	117·7 (2)	O(11)—C(12)—C(13)	111·2 (3)

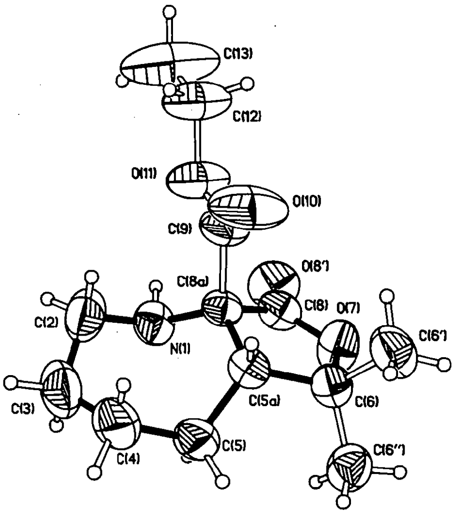

Fig. 1. Thermal-ellipsoid plot of the molecule showing the atom numbering.

298 K, $R = 0·066$ for 1978 observed reflections. The structure was investigated to determine the relative configuration, which could not be established unambiguously by NMR. The perhydroazepine ring adopts a chair conformation.

Experimental. (I): crystal size 0·8 × 0·5 × 0·5 mm. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode involving variable scan width and speed (Clegg, 1981). 2634 reflections measured, $2\theta_{\max}$ 50°, $\pm h + k + l$, three check reflections with no significant intensity change. 2472 unique reflections ($R_{\text{int}} = 0·013$), of which 1978 with $F > 4\sigma(F)$ were used for all calculations (*SHELXS*86, Sheldrick, 1985; *SHELX*76, Sheldrick, 1976). Cell constants refined from $\pm 2\theta$ values of 32 reflections in the range 20–25°. Absorption and extinction corrections were not necessary. Structure solution by direct methods. Refinement on F to $R = 0·066$, $wR = 0·094$; all non-H atoms anisotropic, H atoms were included using a riding model [C—H 0·96 Å, $U(\text{H}) = 0·08$ Å^2, except for methyl protons $U(\text{H}) = 0·12$ Å^2, and $U(\text{H}) = 0·18$ Å^2 for the methyl protons of C(13)], the hydrogen bonded to nitrogen was located in the difference Fourier synthesis and isotropic temperature factor refined. 167 refined parameters, $S = 2·95$, weighting scheme $w^{-1} = \sigma^2(F) + 0·0005F^2$ which led to a featureless analysis of variance in terms of sinθ and F_o, max. $\Delta/\sigma = 0·09$, max. and min. height in final $\Delta\rho$ synthesis 0·27 and −0·36 e Å^{-3} respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

The main reason for the relatively high R index was probably the disordered ethoxycarbonyl group.

Atomic parameters are given in Table 1, selected bond distances and angles in Table 2.* Fig. 1 shows a thermal-ellipsoid plot with the atom numbering.

Related literature. For the preparation of the compound see Tietze & Bratz (1988). For the preparation of some related compounds see Grieco & Fobare (1986).

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* 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 51249 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

We thank the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie for financial support.

References

CLEGG, W. (1981). *Acta Cryst.* A**37**, 22–28.
GRIECO, P. A. & FOBARE, W. F. (1986). *Tetrahedron Lett.* **27**, 5067.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
SHELDRICK, G. M. (1976). *SHELX76*. Crystal structure refinement program. Univ. of Cambridge, England, modified by the author GMS.
SHELDRICK, G. M. (1985). *SHELXS86*. In *Crystallographic Computing* 3, edited by G. M. SHELDRICK, C. KRÜGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.
TIETZE, L.-F. & BRATZ, M. (1988). *Tetrahedron Lett.* Submitted.

Acta Cryst. (1988). C**44**, 2229–2230

Structure of a *D*-Homoestrone Derivative

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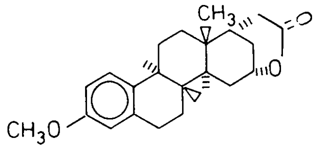
(*Received* 3 *May* 1988; *accepted* 11 *July* 1988)

Abstract. *D*-Homo-3-methoxy-19-nor-17aα-pregna-1,3,5(10)-trien-21,16α-olide, $C_{22}H_{28}O_3$, $M_r = 340{\cdot}46$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}072\ (2)$, $b = 10{\cdot}280\ (2)$, $c = 21{\cdot}618\ (2)$ Å, $V = 1793{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}26$ Mg m^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}08$ mm^{-1}, $F(000) = 736$, $T = 298$ K, $R = 0{\cdot}035$ for 2222 observed reflections. The structure was investigated to determine the constitution of the lactone moiety and the relative configuration, which could not be established unambiguously by NMR. The *B* and *C* rings and the *C* and *D* rings, respectively, are *trans* fused, whereas rings *D* and *E* are *cis*. Rings *C* and *D* adopt chair conformations, whereas rings *B* and *E* show a twisted half-chair and a half-chair conformation respectively.

Experimental. Crystal size 0·5 × 0·6 × 0·7 mm. Stoe–Siemens four-circle diffractometer, monochromated Mo *K*α radiation, profile-fitting mode involving variable scan width and speed (Clegg, 1981). 2853 reflections measured, $2\theta_{\max}$ 50°, h −3→9, k 0→12, l 0→25, three check reflections with no significant intensity change. 2488 unique reflections ($R_{\text{int}} = 0{\cdot}0094$), of which 2222 with $F > 4\sigma(F)$ were used for all calculations [*SHELXS86* (Sheldrick, 1985), *SHELX76* (Sheldrick, 1976)]. Cell constants refined from $\pm 2\theta$ values of 34 reflections in the range 20–25°. Absorption correction was not necessary. Extinction correction was performed yielding a secondary-extinction coefficient x of 0·0020 (5), where $F_c^* = F_c(1 + 0{\cdot}002xF_c^2/\sin 2\theta)^{-0.25}$. Structure solution by direct methods. Refinement on F to $R = 0{\cdot}035$, $wR = 0{\cdot}047$; all non-H atoms anisotropic, H atoms were included using a riding model [C—H 0·96 Å, $U(\text{H}) = 0{\cdot}08$ Å^2, except for methyl protons where $U(\text{H}) = 0{\cdot}12$ Å^2], 227 parameters, $S = 1{\cdot}55$, weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}0005F^2$ which led to a featureless analysis of variance in terms of $\sin\theta$ and F_o, max. $\Delta/\sigma = 0{\cdot}011$, max. and min. height in final $\Delta\rho$ map 0·20 and −0·13 e Å^{-3} respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).



Atomic parameters are given in Table 1, selected bond distances and angles in Table 2.† Fig. 1 shows a thermal-ellipsoid plot with the atom numbering.

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† Lists of structure factors, anisotropic displacement parameters for the non-H atoms and atomic parameters for the H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51228 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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