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Structure of a *D*-Homoestrone Derivative

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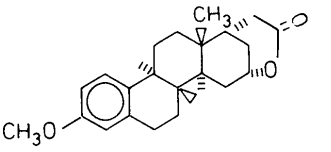
Abstract. *D*-Homo-3-methoxy-19-nor-17aα-pregna-1,3,5(10)-trien-21,16α-olide, $C_{22}H_{28}O_3$, $M_r = 340{\cdot}46$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}072$ (2), $b = 10{\cdot}280$ (2), $c = 21{\cdot}618$ (2) Å, $V = 1793{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}26$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 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 $K\alpha$ 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 ($R_{int} = 0{\cdot}0094$), of which 2222 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 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(H) = 0·08 Å^2, except for methyl protons where U(H) = 0·12 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 sinθ 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.

† 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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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement parameters* ($Å^2 \times 10^3$)

	x	*y*	*z*	U_{eq}*
C(1)	7932 (3)	5867 (2)	4779 (1)	53 (1)
C(2)	8530 (3)	6574 (2)	5276 (1)	56 (1)
C(3)	7532 (3)	6774 (2)	5785 (1)	51 (1)
O(3)	8208 (2)	7501 (1)	6259 (1)	66 (1)
C(3*om*)	7108 (4)	7940 (2)	6726 (1)	72 (1)
C(4)	5961 (3)	6263 (2)	5798 (1)	50 (1)
C(5)	5351 (3)	5543 (2)	5293 (1)	45 (1)
C(6)	3608 (3)	5026 (2)	5338 (1)	55 (1)
C(7)	2878 (2)	4637 (2)	4719 (1)	50 (1)
C(8)	4109 (2)	3814 (2)	4354 (1)	43 (1)
C(9)	5662 (2)	4627 (2)	4208 (1)	44 (1)
C(10)	6340 (3)	5339 (2)	4774 (1)	44 (1)
C(11)	6956 (3)	3799 (2)	3873 (1)	57 (1)
C(12)	6245 (3)	3161 (2)	3293 (1)	58 (1)
C(13)	4669 (2)	2367 (2)	3415 (1)	45 (1)
C(14)	3396 (2)	3242 (2)	3750 (1)	43 (1)
C(15)	1731 (3)	2559 (2)	3823 (1)	56 (1)
C(16)	1096 (3)	2005 (2)	3217 (1)	60 (1)
C(17)	2323 (3)	1107 (2)	2910 (1)	60 (1)
C(18)	3915 (3)	1872 (2)	2798 (1)	52 (1)
C(19)	5128 (3)	1170 (2)	3805 (1)	70 (1)
C(20)	3450 (3)	2915 (2)	2326 (1)	60 (1)
C(21)	1831 (3)	3611 (2)	2430 (1)	61 (1)
O(21*c*)	1495 (3)	4599 (2)	2157 (1)	99 (1)
O(22)	698 (2)	3105 (2)	2809 (1)	67 (1)

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

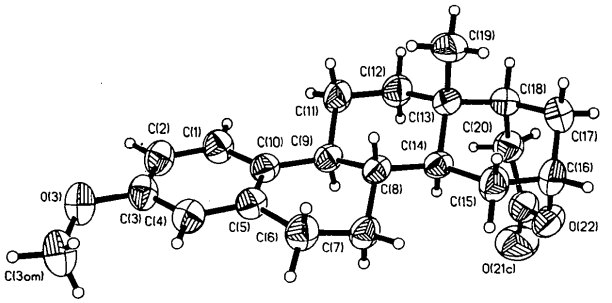

Fig. 1. The asymmetric unit showing 50% thermal displacement ellipsoids.

Related literature. The synthesis of the title compound *via* a tandem-Knoevenagel hetero-Diels–Alder reaction will be published elsewhere (Tietze & Wölfling, 1988); for related reactions see Tietze (1984). For structures of estrone derivatives see Onan, Panda, Cecchini, Chattoraj & Brennan (1981) and Chekhlov, Inov, Ananchenko & Egorova (1984).

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

C(1)–C(2)	1·383 (3)	C(1)–C(10)	1·395 (3)
C(2)–C(3)	1·380 (3)	C(3)–O(3)	1·380 (2)
C(3)–C(4)	1·372 (3)	O(3)–C(3*om*)	1·419 (3)
C(4)–C(5)	1·409 (3)	C(5)–C(6)	1·507 (3)
C(5)–C(10)	1·393 (3)	C(6)–C(7)	1·515 (3)
C(7)–C(8)	1·525 (3)	C(8)–C(9)	1·540 (3)
C(8)–C(14)	1·544 (3)	C(9)–C(10)	1·527 (3)
C(9)–C(11)	1·529 (3)	C(11)–C(12)	1·527 (3)
C(12)–C(13)	1·534 (3)	C(13)–C(14)	1·545 (3)
C(13)–C(18)	1·551 (3)	C(13)–C(19)	1·537 (3)
C(14)–C(15)	1·525 (3)	C(15)–C(16)	1·518 (3)
C(16)–C(17)	1·507 (3)	C(16)–O(22)	1·469 (3)
C(17)–C(18)	1·526 (3)	C(18)–C(20)	1·528 (3)
C(20)–C(21)	1·507 (3)	C(21)–O(21*c*)	1·205 (3)
C(21)–O(22)	1·334 (3)		

C(2)–C(1)–C(10)	122·2 (2)	C(1)–C(2)–C(3)	119·6 (2)
C(2)–C(3)–O(3)	116·2 (2)	C(2)–C(3)–C(4)	119·9 (2)
O(3)–C(3)–C(4)	123·8 (2)	C(3)–O(3)–C(3*om*)	116·9 (2)
C(3)–C(4)–C(5)	120·5 (2)	C(4)–C(5)–C(6)	117·5 (2)
C(4)–C(5)–C(10)	120·2 (2)	C(6)–C(5)–C(10)	122·3 (2)
C(5)–C(6)–C(7)	113·5 (2)	C(6)–C(7)–C(8)	110·5 (2)
C(7)–C(8)–C(9)	109·6 (1)	C(7)–C(8)–C(14)	114·0 (2)
C(9)–C(8)–C(14)	109·7 (1)	C(8)–C(9)–C(10)	112·8 (1)
C(8)–C(9)–C(11)	110·5 (2)	C(10)–C(9)–C(11)	113·7 (2)
C(1)–C(10)–C(5)	117·5 (2)	C(1)–C(10)–C(9)	121·5 (2)
C(5)–C(10)–C(9)	120·8 (2)	C(9)–C(11)–C(12)	111·8 (2)
C(11)–C(12)–C(13)	113·5 (2)	C(12)–C(13)–C(14)	108·8 (2)
C(12)–C(13)–C(18)	110·6 (2)	C(14)–C(13)–C(18)	109·4 (2)
C(12)–C(13)–C(19)	108·7 (2)	C(14)–C(13)–C(19)	111·7 (2)
C(18)–C(13)–C(19)	107·7 (2)	C(8)–C(14)–C(13)	111·7 (1)
C(8)–C(14)–C(15)	114·6 (2)	C(13)–C(14)–C(15)	111·5 (1)
C(14)–C(15)–C(16)	112·4 (2)	C(15)–C(16)–C(17)	112·8 (2)
C(15)–C(16)–O(22)	107·6 (2)	C(17)–C(16)–O(22)	110·6 (2)
C(16)–C(17)–C(18)	107·9 (2)	C(13)–C(18)–C(17)	111·3 (2)
C(13)–C(18)–C(20)	116·2 (2)	C(17)–C(18)–C(20)	105·1 (2)
C(18)–C(20)–C(21)	116·5 (2)	C(20)–C(21)–O(21*c*)	121·5 (2)
C(20)–C(21)–O(22)	120·1 (2)	O(21*c*)–C(21)–O(22)	118·4 (2)
C(16)–O(22)–C(21)	121·3 (2)		

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