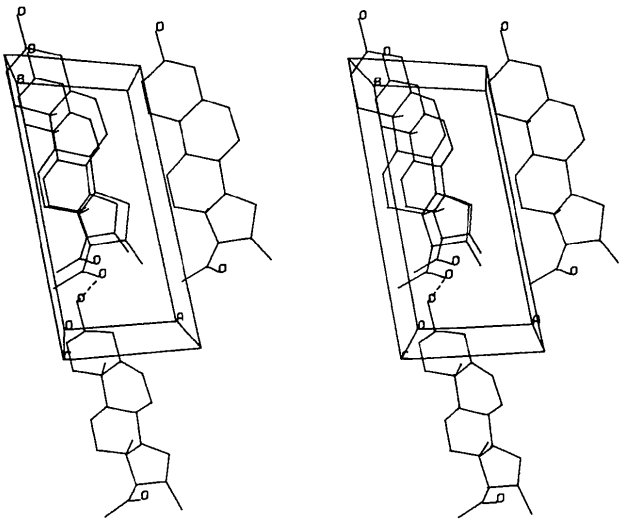

Fig. 2. Stereopacking of the molecule down the *c* axis, showing the hydrogen bonding.

map and refined with isotropic temperature parameters. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Final $R = 0.043$, $wR = 0.050$, $S = 1.210$ for observed reflections and $R = 0.048$ for all data, $w = 1/\sigma^2$, $(\Delta/\sigma)_{max} = 0.19$. Weighting scheme based on estimates of experimental errors from counting statistics. Final difference map showed maximum positive and negative peaks of $+0.42$ and -0.15 e Å^{-3}. No corrections for absorption or extinction were made.

Atomic parameters are listed in Table 1. Distances, angles and selected torsion angles are listed in Table 2.* A stereoscopic view of the molecule showing the atomic numbering and molecular conformation is given in Fig. 1. Fig. 2 shows a stereoview of the molecular packing.

Related literature. This structure is one of a series of steroid structures related to progesterone. Although 16β-substitution has been shown to destabilize the minimum energy conformation of the progesterone side chain, 16α-substitution does not have the same effect. The C16—C17—C20—O20 torsion angle, $-29.4°$, is in the center of the observed range for 81 of 85 crystallographically observed pregnanes (Duax, Griffin & Rohrer, 1981).

* Lists of structure factors, anisotropic displacement parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51890 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 7,10-Dimethoxy-2,6,6,9-tetramethyltricyclo[5.4.0.0^{1,5}]-undec-9-ene-8,11-dione*

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Abstract. $C_{17}H_{24}O_4$, $M_r = 292.4$, monoclinic, $P2_1$, $a = 7.937$ (3), $b = 14.031$ (6), $c = 8.143$ (3) Å, $\beta = 116.59$ (3)°, $V = 811$ (1) Å^3, $Z = 2$, $D_x = 1.20$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 0.078$ mm^{-1}, $F(000) = 316$, $T = 293$ K, $R = 0.057$ for 780 observed reflections. The X-ray study confirms that in the solid state the structure of the title compound is similar to that inferred from chemical and spectroscopic evidence. The Cremer & Pople [*J. Am. Chem. Soc.* (1975), **97**, 1354–1358] ring-puckering parameters for the six-membered *A* ring and five-membered *C* ring are $\theta = 99$ (1), $\varphi = 120$ (1)°, $Q = 0.298$ (8) Å, and $\varphi = -119$ (1)°, $Q =$

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0108-2701/89/091435-03$03.00

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

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

	x	y	z	U_{eq}
O(1)	5873 (6)	4972	2269 (7)	78 (3)
O(2)	12887 (7)	5963 (4)	4819 (7)	87 (3)
O(3)	8585 (6)	3400 (4)	3500 (7)	72 (2)
O(4)	10473 (6)	6469 (4)	1175 (7)	76 (3)
C(1)	11090 (8)	4583 (5)	4759 (8)	45 (3)
C(2)	12411 (10)	3792 (6)	4698 (10)	68 (4)
C(3)	12601 (11)	3134 (7)	6253 (11)	93 (4)
C(4)	12640 (11)	3791 (7)	7712 (11)	92 (4)
C(5)	11374 (10)	4619 (6)	6731 (9)	67 (4)
C(6)	9253 (11)	4580 (6)	6127 (11)	73 (4)
C(7)	8949 (9)	4348 (6)	4050 (9)	56 (4)
C(8)	7514 (9)	5003 (6)	2602 (8)	53 (3)
C(9)	8176 (10)	5647 (6)	1566 (9)	56 (3)
C(10)	10001 (8)	5848 (5)	2152 (9)	52 (3)
C(11)	11458 (8)	5496 (5)	3988 (9)	53 (3)
C(12)	6796 (10)	3014 (7)	2788 (12)	98 (5)
C(13)	12308 (10)	6452 (8)	1256 (12)	93 (5)
C(14)	11984 (11)	3352 (7)	2838 (12)	93 (5)
C(15)	8589 (13)	3805 (8)	6992 (11)	104 (5)
C(16)	8465 (13)	5549 (7)	6265 (11)	97 (5)
C(17)	6644 (10)	6124 (6)	−86 (10)	74 (4)

0·360 (8) Å indicating boat and envelope conformations, respectively. The four-membered *B* ring, with a puckering angle of 13·5 (6)°, is fused to the five- and six-membered rings on adjacent edges and exhibits bond lengths in the range 1·519 (11)–1·631 (12) Å. The *A*/*B* and *B*/*C* junctions are *cis*. An intermolecular C—H···O contact $<3·4$ Å is present: C(13)···O(1) (1 = *x*, *y*, *z*) = 3·30 (1) Å. The molecules in the crystal are packed at normal van der Waals distances.

Experimental. The title compound was recrystallized from pentane and gave pale-yellow crystals, m.p. 357–358 K. Crystal size 0·22 × 0·28 × 0·24 mm. Nicolet *R*3 four-circle diffractometer. Unit-cell parameters by least-squares refinement from 25 machine-centred reflections with $4·1 < 2\theta < 17·2°$. 1112 unique reflections measured for two octants, $3 < 2\theta < 45°$, 780 with $I > 2·5\sigma(I)$ used in analysis, index range $h \pm 7$, $k\ 0 \rightarrow 14$, $l\ 0 \rightarrow 8$, $R_{int} = 0·030$, ω-scan mode, scan width 1·0°(θ). Two standard reflections ($0\bar{2}0$, 001) monitored every 50 measurements, no significant variation. Intensities corrected for Lorentz–polarization but not for absorption. Data adjusted to an approximately absolute scale and an overall U value of 0·050 $Å^2$. Structure solved by combination of direct methods and partial structure expansion by an iterative *E*-Fourier procedure using *SHELXTL5* (Sheldrick, 1985). Least-squares refinement of all non-H atoms anisotropic; H atoms in calculated positions riding on the bonded C with a fixed isotropic temperature factor, $U = 0·06$ $Å^2$. $\sum w(\Delta F)^2$ minimized, $w = [\sigma^2(F_o) + 0·001(F_o)^2]^{-1}$, where σ is standard deviation of observed amplitudes, based on counting statistics;

Table 2. *Molecular geometry*

E.s.d.'s are given in parentheses.

(*a*) Bond lengths (Å)

O(1)—C(8)	1·207 (9)	O(2)—C(11)	1·220 (8)
O(3)—C(7)	1·391 (10)	O(3)—C(12)	1·382 (9)
O(4)—C(10)	1·341 (10)	O(4)—C(13)	1·428 (10)
C(1)—C(2)	1·542 (11)	C(1)—C(5)	1·519 (11)
C(1)—C(7)	1·566 (9)	C(1)—C(11)	1·512 (11)
C(2)—C(3)	1·519 (13)	C(2)—C(14)	1·526 (13)
C(3)—C(4)	1·493 (14)	C(4)—C(5)	1·510 (11)
C(5)—C(6)	1·529 (12)	C(6)—C(7)	1·631 (12)
C(6)—C(15)	1·512 (15)	C(6)—C(16)	1·521 (13)
C(7)—C(8)	1·527 (9)	C(8)—C(9)	1·483 (12)
C(9)—C(10)	1·337 (10)	C(9)—C(17)	1·506 (9)
C(10)—C(11)	1·506 (8)		

(*b*) Valence angles (°)

C(7)—O(3)—C(12)	122·5 (6)	C(10)—O(4)—C(13)	120·7 (6)
C(2)—C(1)—C(5)	106·1 (5)	C(2)—C(1)—C(7)	118·6 (6)
C(5)—C(1)—C(7)	91·1 (6)	C(2)—C(1)—C(11)	109·6 (7)
C(5)—C(1)—C(11)	116·5 (6)	C(7)—C(1)—C(11)	113·9 (5)
C(1)—C(2)—C(3)	103·0 (7)	C(1)—C(2)—C(14)	118·1 (5)
C(3)—C(2)—C(14)	118·4 (8)	C(2)—C(3)—C(4)	104·3 (7)
C(3)—C(4)—C(5)	106·4 (6)	C(1)—C(5)—C(4)	106·4 (7)
C(1)—C(5)—C(6)	92·2 (5)	C(4)—C(5)—C(6)	121·2 (7)
C(5)—C(6)—C(7)	88·3 (6)	C(5)—C(6)—C(15)	116·0 (6)
C(7)—C(6)—C(15)	114·7 (7)	C(5)—C(6)—C(16)	111·9 (7)
C(7)—C(6)—C(16)	112·3 (6)	C(15)—C(6)—C(16)	111·6 (9)
O(3)—C(7)—C(1)	110·8 (6)	O(3)—C(7)—C(6)	116·0 (7)
C(1)—C(7)—C(6)	86·8 (5)	O(3)—C(7)—C(8)	110·6 (5)
C(1)—C(7)—C(8)	118·0 (7)	C(6)—C(7)—C(8)	113·2 (6)
O(1)—C(8)—C(7)	120·6 (7)	O(1)—C(8)—C(9)	121·0 (6)
C(7)—C(8)—C(9)	118·4 (6)	C(8)—C(9)—C(10)	121·9 (6)
C(8)—C(9)—C(17)	115·4 (6)	C(10)—C(9)—C(17)	122·5 (8)
O(4)—C(10)—C(9)	118·1 (6)	O(4)—C(10)—C(11)	120·5 (6)
C(9)—C(10)—C(11)	120·9 (7)	O(2)—C(11)—C(1)	122·0 (6)
O(2)—C(11)—C(10)	119·0 (7)	C(1)—C(11)—C(10)	119·0 (5)

(*c*) Selected torsion angles (°)

C(11)—C(1)—C(7)—C(8)	4·2 (10)	C(5)—C(1)—C(7)—C(6)	9·4 (6)
C(7)—C(1)—C(5)—C(6)	−10·0 (6)	C(2)—C(1)—C(5)—C(4)	−6·7 (8)
C(13)—O(4)—C(10)—C(9)	−155·8 (8)	O(1)—C(8)—C(9)—C(17)	−9·3 (1)
O(3)—C(7)—C(8)—O(1)	65·6 (9)		

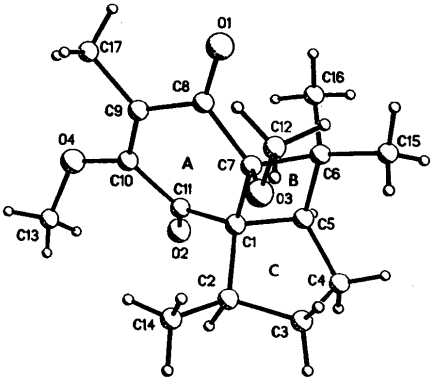

Fig. 1. The molecular structure of the title compound with atom numbering.

isotropic extinction parameter $X = 0·002$. In the last cycle $(\Delta/\sigma)_{max} = 0·10$; $\Delta\rho$ from −0·18 to 0·17 e $Å^{-3}$, $S = 1·16$; final $R = 0·057$, $wR = 0·059$; 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.

Atomic coordinates are given in Table 1.* A perspective molecular drawing and the atom labelling are shown in Fig. 1. Bond distances, angles and selected torsion angles are listed in Table 2.

Related literature. The title compound was prepared as part of an investigation on the ultraviolet irradiation of *O*-methylmethoxyperezone (Barrera, Barrios & Walls, 1980). Its chemical structure was formulated from chemical and spectroscopic evidence (Barrios, Salazar, Diaz, Walls & Joseph-Nathan, 1986).

We thank Mrs Cynthia Lesh de S. for technical assistance.

* Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51877 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 9,11-Dimethoxy-2,6,7-trimethyltetracyclo-[4.3.3.0^{1,5}.0^{7,11}]dodecane-8,10-dione*

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Abstract. $C_{17}H_{24}O_4$, $M_r = 292{\cdot}4$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}376$ (1), $b = 13{\cdot}612$ (6), $c = 15{\cdot}551$ (4) Å, $V = 1561$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}24$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}7107$ Å, $\mu = 0{\cdot}082$ mm^{-1}, $F(000) = 632$, $T = 293$ K, $R = 0{\cdot}061$ for 832 observed reflections. The stereochemistry of the title compound is C(5)—αH *trans* to C(6)—βCH$_3$, C(8)—βO(1) *trans* to C(9)—α(methoxy), C(10)—αO(3) *cis* to C(11)—α(methoxy) with a *cis, anti, anti* relationship of methyl groups at C(6), C(7); C(6), C(13); and C(7), C(13), respectively. The four- and five-membered rings adopt puckered [38·9 (5)°] and half-chair conformations, respectively. Both six-membered rings [C(1)—C(5)—C(6)—C(7)—C(8)—C(9) and C(1)—C(10)—C(11)—C(7)—C(8)—C(9)] exhibit a boat conformation. The four-membered ring has three long bonds [1·580 (11)–1·601 (10) Å] and one short bond at 1·532 (10) Å. A C—H···O intermolecular contact is present, C(16)···O(3)($-\frac{1}{2}+x, \frac{1}{2}-y, -z$) 3·25 (1) Å. The packing in the crystal is entirely due to van der Waals forces.

Experimental. The title compound was recrystallized from acetone–ethanol and gave colourless crystals, m.p. 427–428 K. Crystal size 0·15 × 0·32 × 0·32 mm. Nicolet *R*3 four-circle diffractometer. Unit-cell parameters by least-squares refinement from 25 machine-centred reflections with $4{\cdot}5 < 2\theta < 18{\cdot}5°$. 1204 unique reflections measured for an octant, $3 < 2\theta < 45°$, of which 832 with $I > 2{\cdot}5\sigma(I)$ were used in the analysis. Index range h 0→7, k 0→14, l 0→16, ω-scan mode and variable scan speed. Two standard reflections ($\bar{1}0\bar{3}$, $\bar{1}10$) monitored every 50 measurements; no significant variation. Lp correction, absorption ignored and $R_{int} = 0{\cdot}012$. Structure solved by direct methods using *SHELXTL5* (Sheldrick, 1985). Least-squares refinement of all non-H atoms with anisotropic thermal parameters; H atoms in calculated positions riding on the bonded C with a

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