the formation of hydrogen bonds. As would be expected, both hydroxyl groups in 11β-hydroxy-9β-estrone form hydrogen bonds [O(3)→O(11β) = 2·80 Å, O(3)—H···O(11β) = 131·5°; O(11β)→O(17) = 2·82 Å, O(11β)—H···O(17) = 176·1°] in spite of the fact that the hydrogen-bond angle in one is far from linear. The lone hydrogen bond in 11-keto-9β-estrone [O(3)→O(17) = 2·80 Å, O(3)—H···O(17) = 166·1°] is of the head-to-tail type most commonly observed in steroid structures (Duax & Norton, 1975). The 11β-hydroxyl group disrupts the normal head-to-tail pattern and acts as a donor and an acceptor of hydrogen bonds (Fig. 3). This difference in intramolecular interaction between the 11β-hydroxy group and the 11-oxo atom which accepts no hydrogen bonds and has only normal van der Waals contacts with its surroundings is the most significant difference noted between the structures.

Since the overall shapes and relative positions of the O(3) and O(17) substituents are nearly identical in 11β-hydroxy-9β-estrone and 11-keto-9β-estrone, the activity difference between the two is likely to be a direct consequence of molecular interaction of their C(11) substituents. The fact that the 11β-hydroxy substrate forms strong hydrogen bonds and disrupts normal patterns in steroid association in the solid state may indicate that comparable hydrophilic and hydrogen-bonding interactions interfere with the ability of the compound to function as a potent estrogen.

A sample of the title compound was generously provided by the late Dr A. Segaloff. This work was
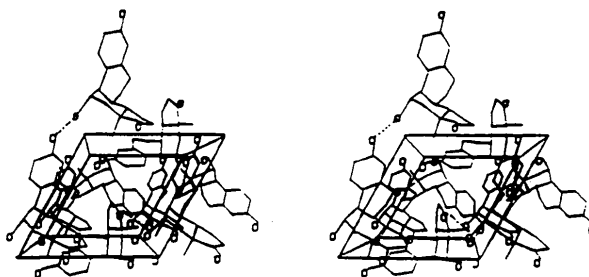

Fig. 3. Stereoview of the crystal structure of 11β-hydroxy-9β-estrone viewed down the c axis.

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Functionalized Macrocarbocyclic Rings. I. Two Fourteen-Membered Dimethyl Macrocycles with *trans, cis* Diene and *cis* Dienophile

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Abstract. $C_{24}H_{34}O_8$, $M_r = 450·53$, $D_x = 1·23$ Mg m⁻³, $\lambda(Cu\,K\bar{\alpha}) = 1·5418$ Å, room temperature. (I) Tetra- methyl (3Z,5E,11Z)-3,11-dimethylcyclotetradeca-3,5,-11-triene-1,1,8,8-tetracarboxylate, monoclinic, $P2_1/c$, $a = 16·025$ (5), $b = 9·919$ (4), $c = 16·997$ (8) Å, $\beta = 115·60$ (4)°, $V = 2436·49$ Å³, $Z = 4$, $\mu =$

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0.720 mm^{-1}, $F(000) = 968$, final $R = 0.049$ for 2805 observed reflections. (II) Tetramethyl $(3Z,5E,11Z)$-3,12-dimethylcyclotetradeca-3,5,11-triene-1,1,8,8-tetra-carboxylate, triclinic, $P\bar{1}$, $a = 12.265\,(2)$, $b = 15.756\,(3)$, $c = 7.051\,(2)$ Å, $\alpha = 99.71\,(1)$, $\beta = 105.01\,(2)$, $\gamma = 106.86\,(1)^\circ$, $V = 1214.94$ Å^3, $Z = 2$, $\mu = 0.722$ mm^{-1}, $F(000) = 484$, final $R = 0.051$ for 2848 observed reflections. Fourteen-membered macro-carbocycles (I) and (II) each contain two pairs of methyl esters borne on C atoms C(5) and C(12) in addition to a methyl group at C(7). A second methyl group is carried on C(1) in (I) and on C(2) in (II). The double bonds $\Delta^{9,10}$, $\Delta^{7,8}$ and $\Delta^{1,2}$ are *trans, cis* and *cis* respectively for both macrocycles. Torsion angles indicate that the diene portion lies roughly in a plane. Two *gauche* interactions are found at the junctions of this plane with the two roughly *anti* arrangements of C-atom chains C(2)–C(3)–C(4)–C(5) and C(12)–C(13)–C(14)–C(1) that rise up to the *cis* olefin. Support for the synthetic strategy involving trans-annular Diels–Alder cycloadditions of (I) and (II) is provided by this study.

Introduction. Intramolecular reactions play an increasingly large role in organic synthesis today. For example, some recent communications from these laboratories have detailed the relative stereochemistries and conformational properties of tricyclic compounds which result from the transannular Diels–Alder reaction of trienic macrocycles (Michel, Proulx & Michel-Dewez, 1988; Michel, Boulay & Marinier, 1989; Michel, Michel-Dewez & Roughton, 1989; Michel, Michel-Dewez, Roughton, Springer & Hoogsteen, 1989).

Such a transannular strategy for the construction of polycyclic molecules (Lamothe, Ndibwami & Deslong-champs, 1988a; Mariner & Deslongchamps, 1988) involves the traditionally difficult synthesis of large rings. Internal processes in which open chains of atoms become closed rings are aided by the presence of unsaturation points which diminish both the number of degrees of freedom and the steric transannular inter-actions (Deslongchamps, 1984). Thus, the triene precursors to (I) and (II) (Fig. 1) were cyclized to their respective macrocycles by S_{N^2} displacement of the allylic chlorides with the caesium-carbonate-generated enolate ions. This strategy regulates two problems at once: the difficult step of macrocyclization is made easier by the three points of unsaturation which, in addition, provide subsequent opportunity for specific transannular cycloadditions.

The conformational properties of a macrocycle change with each olefinic geometry combination. Moreover, predictions and empirical evidence show (Lamothe, Ndibwami & Deslongchamps, 1988a,b) that the relative stereochemistry of the four asymmetric centres in a cyclo-addition adduct can be controlled by

macrocyclic olefin geometries. Intramolecular processes therefore depend on both olefinic combinations and resulting macrocycle conformations, accentuating the necessity for X-ray analysis.

The present crystallographic study of macrocycles (I) and (II) was undertaken in the hope that the results from the combined X-ray data for macrocycles and tricycles would validate the potential of the methodology underlying their syntheses.

Experimental. Crystals $0.25 \times 0.1 \times 0.1$ mm, automatic four-circle diffractometer with Cu $K\bar{\alpha}$ radiation; lattice parameters determined from 25 medium-angle $(60 \le 2\theta \le 100^\circ)$ reflections; one standard reflection monitored every 100 reflections without significant deviation.
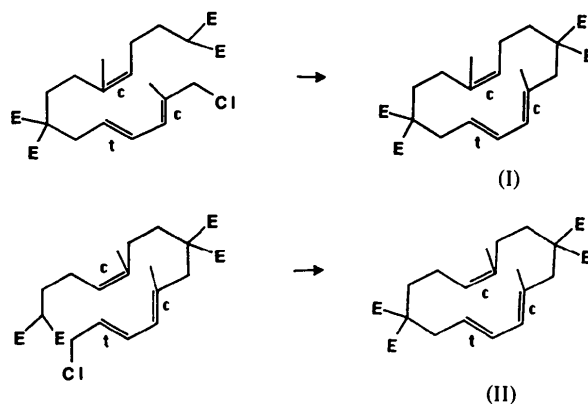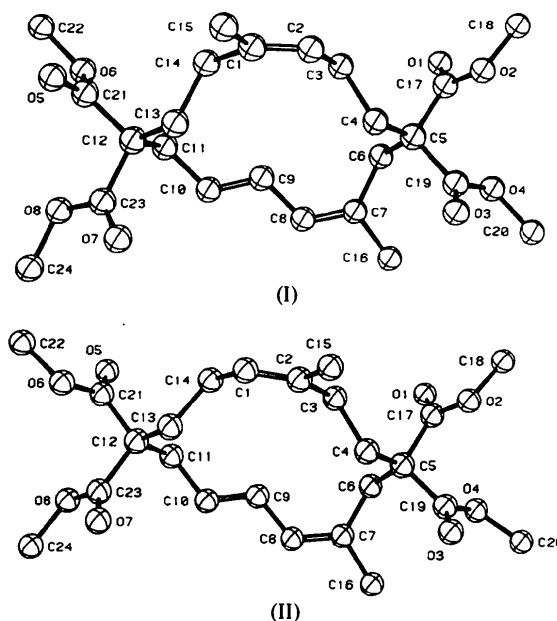

Fig. 1. Synthetic scheme. $E = COOCH_3$.

Fig. 2. Perspective view for both title compounds.

Table 1. *Final coordinates and B_{iso} values* ($\text{Å}^2 \times 10^2$), *with e.s.d.'s in parentheses, of compounds* (I) *and* (II)

$$B_{iso} = \tfrac{8}{3}\pi^2 \text{ trace}(U).$$

	Compound (I)				Compound (II)			
	x	y	z	B_{iso}	x	y	z	B_{iso}
O(1)	−0·0794 (1)	0·3701 (1)	0·8793 (1)	5·26	0·6345 (1)	0·2834 (1)	0·6186 (3)	6·40
O(2)	0·0199 (1)	0·2171 (1)	0·9663 (1)	4·59	0·7006 (1)	0·3660 (1)	0·4178 (3)	5·23
O(3)	0·0268 (1)	−0·0312 (2)	0·8278 (1)	7·65	0·8105 (1)	0·2408 (1)	0·1380 (3)	5·01
O(4)	0·0544 (1)	0·1824 (2)	0·8068 (1)	6·13	0·6247 (1)	0·1781 (1)	0·1523 (2)	4·42
O(5)	−0·5981 (1)	−0·2343 (2)	0·7378 (1)	6·47	1·1844 (2)	0·2643 (2)	1·6120 (3)	7·90
O(6)	−0·6282 (1)	−0·0344 (2)	0·6738 (1)	6·31	1·3641 (2)	0·3290 (1)	1·5855 (3)	5·46
O(7)	−0·4578 (1)	−0·3540 (2)	0·6185 (1)	7·15	1·3535 (2)	0·1964 (1)	1·1318 (3)	8·10
O(8)	−0·5981 (1)	−0·2646 (2)	0·5473 (1)	5·91	1·3036 (1)	0·1267 (1)	1·3623 (3)	5·83
C(1)	−0·2994 (1)	−0·1044 (2)	0·9112 (1)	4·16	1·1494 (2)	0·4297 (2)	1·0499 (4)	3·94
C(2)	−0·2203 (1)	−0·0387 (2)	0·9349 (1)	4·40	1·0715 (2)	0·4300 (1)	0·8818 (3)	3·63
C(3)	−0·1965 (1)	0·0757 (2)	0·8903 (1)	4·49	0·9431 (2)	0·3630 (2)	0·7869 (4)	3·99
C(4)	−0·1240 (1)	0·0308 (2)	0·8598 (1)	4·31	0·9114 (2)	0·3117 (2)	0·5644 (4)	3·72
C(5)	−0·0811 (1)	0·1422 (2)	0·8257 (1)	4·06	0·7784 (2)	0·2482 (2)	0·4660 (3)	3·52
C(6)	−0·1486 (1)	0·2000 (2)	0·7357 (1)	4·41	0·7502 (2)	0·1594 (2)	0·5419 (3)	3·60
C(7)	−0·1751 (1)	0·1072 (2)	0·6588 (1)	5·22	0·8163 (2)	0·0964 (2)	0·4955 (4)	3·66
C(8)	−0·2562 (1)	0·0425 (2)	0·6216 (1)	4·88	0·9023 (2)	0·0841 (2)	0·6397 (4)	4·02
C(9)	−0·3331 (1)	0·0479 (2)	0·6443 (1)	4·49	0·9454 (2)	0·1266 (2)	0·8564 (4)	4·00
C(10)	−0·4080 (1)	−0·0291 (2)	0·6081 (1)	4·55	1·0364 (2)	0·1164 (2)	0·9896 (4)	4·31
C(11)	−0·4880 (1)	−0·0239 (2)	0·6302 (1)	4·22	1·0822 (2)	0·1613 (2)	1·2109 (4)	4·34
C(12)	−0·4976 (1)	−0·1538 (2)	0·6768 (1)	4·02	1·2111 (3)	0·2339 (2)	1·2826 (4)	3·82
C(13)	−0·4098 (1)	−0·1828 (2)	0·7609 (1)	4·36	1·2192 (2)	0·3084 (2)	1·1644 (4)	3·92
C(14)	−0·3867 (1)	−0·0740 (2)	0·8305 (1)	4·18	1·1353 (2)	0·3624 (2)	1·1772 (4)	4·08
C(15)	−0·3082 (2)	−0·2190 (2)	0·9647 (1)	5·62	1·1058 (2)	0·5040 (2)	0·7757 (4)	4·94
C(16)	−0·1074 (2)	0·0982 (4)	0·6188 (2)	8·38	0·7794 (3)	0·0402 (2)	0·2802 (4)	5·26
C(17)	−0·0488 (1)	0·2578 (2)	0·8912 (1)	4·21	0·6959 (2)	0·2992 (2)	0·5132 (4)	3·73
C(18)	0·0602 (2)	0·3160 (2)	1·0339 (1)	5·71	0·6176 (2)	0·4132 (2)	0·4268 (5)	5·89
C(19)	0·0054 (1)	0·0845 (2)	0·8207 (1)	4·79	0·7435 (2)	0·2232 (2)	0·2342 (3)	3·77
C(20)	0·1344 (2)	0·1404 (3)	0·7945 (2)	7·64	0·5730 (3)	0·1568 (2)	−0·0654 (4)	5·76
C(21)	−0·5801 (1)	−0·1473 (2)	0·6989 (1)	4·19	1·2486 (2)	0·2764 (2)	1·5097 (4)	4·60
C(22)	−0·7052 (2)	−0·0208 (3)	0·6961 (2)	9·01	1·4133 (3)	0·3656 (3)	1·8047 (5)	7·33
C(23)	−0·5143 (2)	−0·2703 (2)	0·6127 (1)	4·98	1·2985 (2)	0·1856 (2)	1·2481 (4)	4·05
C(24)	−0·6197 (2)	−0·3604 (3)	0·4772 (2)	7·16	1·3811 (2)	0·0753 (2)	1·3410 (5)	6·59

(I) $-15 \le h \le 6$, $-9 \le k \le 10$, $-18 \le l \le 16$; 3279 unique measured reflections, 2805 observed with $I \ge 3\sigma(I)$; $2\theta_{max} = 144°$; $R = 0·049$, $wR = 0·071$; max. Δ/σ (for non-H atoms) = 0·32; max. and min. density peak = 0·15 and −0·12 e Å^{-3}.

(II) $0 \le h \le 13$, $-17 \le k \le 16$, $-7 \le l \le 7$; 3274 unique measured reflections, 2848 observed with $I \ge 3\sigma(I)$; $2\theta_{max} = 144°$; $R = 0·051$, $wR = 0·088$; max. Δ/σ (for non-H atoms) = 0·23; max. and min. density peak = 0·18 and −0·10 e Å^{-3}.

For both structures, H atoms were assigned isotropic temperature factors corresponding to their attached atoms. The function $\sum w(|F_o| - |F_c|)^2$ with $w = 1/(\sigma F_o)^2$ was minimized. The structures were solved by direct methods with *SHELXS*86 (Sheldrick, 1986), difference Fourier analysis and refinement using full-matrix least-squares techniques *SDP-Plus* VI.1 (Okaya & Frenz, 1984). No abnormally short intermolecular contacts were noted. Atomic scattering factors and f', f'' values from *SHELXS*86.

Discussion. Final atomic parameters are given in Table 1.* Bond lengths, valence angles and selected torsion

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51674 (37 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

angles are given in Table 2. Fig. 1 shows the synthetic scheme. Perspective views of both title compounds are depicted in Fig. 2.

The results of the structure determination indicate clearly that (I) and (II) are fourteen-membered carbocyclic rings having identical olefin geometries in which the double bonds $\Delta^{9,10}$, $\Delta^{7,8}$ and $\Delta^{1,2}$ are *trans, cis* and *cis* respectively (Fig. 2). Both macrocycles bear a methyl group on C(7); however, the second methyl group of (I) is borne on C(1) while that of (II) is borne on C(2). Pairs of methyl esters are carried on C(5) and C(12). All bond lengths and bond angles appear to be relatively normal.

Great similarities exist between the conformational properties of (I) and (II). In general the carbonyl groups of the pairs of esters are antiperiplanar to one another. The curious exceptions to this observation are the carbonyl groups of the C(12) esters in (I). All esters exist in the more stable Z form (Deslongchamps, 1983). Interesting information about the conformations of the macrocycles is found especially in the torsion angles. For example, the angles formed by C(12)—C(13)—C(14)—C(1) and by C(2)—C(3)—C(4)—C(5) are in the order of −170·8 to −178·9° for both (I) and (II) illustrating the relative planarity of these low energy *anti* forms of *n*-butane. The total planarity/conjugation of the diene sections of (I) and (II) is slightly broken as evidenced by the average torsion angle of −174·6° for C(7)—C(8)—C(9)—C(10). The double bonds of (I) and

Table 2. Molecular geometry

(a) Intramolecular bond lengths (Å) with e.s.d.'s in parentheses

	(I)	(II)		(I)	(II)
O(1)-C(17)	1·1985 (22)	1·194 (4)	C(3)-C(4)	1·529 (3)	1·530 (4)
O(2) C(17)	1·3379 (20)	1·337 (4)	C(4)-C(5)	1·541 (3)	1·542 (3)
O(2) C(18)	1·4347 (22)	1·432 (4)	C(5)-C(6)	1·5521 (19)	1·559 (4)
O(3) C(19)	1·189 (3)	1·197 (3)	C(5)-C(17)	1·5245 (20)	1·528 (4)
O(4) C(19)	1·333 (3)	1·3367 (23)	C(5)-C(19)	1·536 (4)	1·527 (3)
O(4) C(20)	1·447 (4)	1·438 (3)	C(6)-C(7)	1·502 (3)	1·498 (5)
O(5) C(21)	1·196 (3)	1·196 (4)	C(7) C(8)	1·3381 (23)	1·347 (4)
O(6) C(21)	1·321 (3)	1·325 (3)	C(7)-C(16)	1·511 (4)	1·501 (4)
O(6) C(22)	1·445 (4)	1·449 (4)	C(8)-C(9)	1·443 (3)	1·447 (4)
O(7)- C(23)	1·201 (3)	1·193 (4)	C(9)-C(10)	1·3283 (23)	1·329 (4)
O(8) C(23)	1·324 (3)	1·332 (4)	C(10)-C(11)	1·484 (3)	1·481 (4)
O(8) C(24)	1·444 (4)	1·436 (4)	C(11)-C(12)	1·555 (3)	1·551 (3)
C(1) C(2)	1·3238 (24)	1·317 (3)	C(12)-C(13)	1·5387 (20)	1·545 (4)
C(1) C(14)	1·5071 (20)	1·502 (5)	C(12)-C(21)	1·524 (3)	1·517 (3)
C(1) C(15)	1·500 (3)	—	C(12)-C(23)	1·530 (3)	1·527 (4)
C(2) C(3)	1·503 (3)	1·515 (3)	C(13)-C(14)	1·524 (3)	1·523 (4)
C(2) C(15)	—	1·506 (4)			

(b) Valence angles (°) with e.s.d.'s in parentheses

	(I)	(II)
C(17) O(2) C(18)	117·25 (14)	117·18 (23)
C(19) -O(4)-C(20)	116·28 (20)	149·5 (4)
C(21)-O(6)-C(22)	116·07 (20)	116·5 (3)
C(23) O(8) -C(24)	117·34 (20)	116·44 (24)
C(2) C(1)-C(14)	124·66 (17)	128·85 (20)
C(2)-C(1)-C(15)	121·14 (15)	—
C(14)-C(1)-C(15)	114·20 (16)	—
C(1)-C(2)-C(3)	129·54 (14)	124·95 (22)
C(1) C(2)-C(15)	—	119·94 (19)
C(3) C(2) -C(15)	—	115·03 (20)
C(2)-C(3)-C(4)	110·29 (16)	114·19 (22)
C(3) -C(4)-C(5)	116·53 (17)	113·61 (22)
C(4)-C(5)-C(6)	113·67 (18)	112·53 (22)
C(4) C(5) C(17)	109·30 (14)	110·14 (22)
C(4)- C(5)-C(19)	108·28 (13)	110·55 (21)
C(6) C(5)--C(17)	108·30 (12)	108·35 (22)
C(6) C(5)--C(19)	110·15 (15)	110·29 (21)
C(17) C(5)- C(19)	106·92 (18)	104·67 (22)
C(5) C(6) C(7)	116·48 (15)	116·26 (23)
C(6) C(7)-C(8)	123·83 (18)	123·03 (24)
C(6)- C(7)- C(16)	116·08 (18)	118·25 (24)
C(8) C(7) C(16)	119·90 (19)	118·6 (3)
C(7)-C(8)-C(9)	128·56 (17)	127·4 (3)
C(8)--C(9)-C(10)	124·06 (18)	124·6 (3)
C(9) C(10)- C(11)	124·69 (18)	124·6 (3)
C(10)- C(11)- C(12)	112·74 (15)	113·11 (23)
C(11) C(12) -C(13)	112·10 (13)	111·96 (20)
C(11)--C(12)-C(21)	112·21 (15)	108·87 (21)
C(11)--C(12)-C(23)	106·77 (15)	108·85 (23)
C(13)-C(12)-C(21)	108·75 (15)	111·09 (23)
C(13)- C(12)--C(23)	109·19 (16)	108·48 (22)
C(21)- C(12)-C(23)	107·70 (17)	107·46 (21)
C(12) C(13) C(14)	113·99 (15)	115·80 (24)
C(1)- C(14)--C(13)	112·55 (15)	110·11 (25)
O(1)-C(17)-O(2)	123·33 (16)	123·2 (3)
O(1) -C(17)-C(5)	126·21 (13)	126·5 (3)
O(2)- C(17)-C(5)	110·46 (15)	110·33 (23)
O(3)- C(19)- O(4)	123·73 (19)	124·39 (18)
O(3) C(19)--C(5)	125·58 (18)	126·22 (19)
O(4)- C(19) -C(5)	110·69 (16)	109·39 (20)
O(5) C(21)--O(6)	122·94 (20)	122·84 (23)
O(5) C(21) C(12)	123·23 (18)	125·52 (19)
O(6) C(21) ·C(12)	113·79 (17)	111·63 (24)
O(7) C(23)· O(8)	123·63 (18)	123·5 (3)
O(7)- C(23) ·C(12)	124·75 (18)	125·6 (3)
O(8) C(23) C(12)	111·56 (21)	110·89 (23)

(c) Selected torsion angles (°) with e.s.d.'s in parentheses

	(I)	(II)
C(14) C(1)· C(2)·-C(3)	-0·9 (1)	4·6 (1)
C(1) C(2) C(3) ·C(4)	114·6 (2)	-124·9 (2)
C(3) C(4) C(5) C(6)	-71·4 (1)	73·8 (2)
C(5)- C(6) C(7) C(8)	102·5 (2)	110·6 (2)
C(7)- C(8)-C(9)-C(10)	-174·0 (2)	-175·2 (3)
C(9)--C(10) -C(11) C(12)	-112·4 (2)	112·7 (2)
C(11) C(12) C(13) C(14)	62·7 (1)	58·4 (1)
C(2) C(1) C(14)--C(13)	108·9 (2)	112·5 (2)
C(2) C(3) C(4) C(5)	170·8 (2)	-176·5 (2)
C(4) C(5) C(6) C(7)	-68·9 (1)	62·2 (1)
C(6) C(7) C(8)--C(9)	1·0 (1)	0·3 (1)
C(8) C(9) -C(10)--C(11)	-179·4 (2)	178·7 (2)
C(10) C(11) C(12)--C(13)	57·4 (1)	56·1 (1)
C(12) C(13) -C(14)--C(1)	-178·9 (2)	-178·0 (2)

(II) are almost completely planar ($\pm 1°$) with the exception of the isolated cis olefin of (II) which has a 4·6° torsion angle for C(14)—C(1)—C(2)—C(3).

In qualitative terms, the diene forms a lower plane from which rise the two planar chains leading to the cis olefin (Fig. 2). In order to avoid the strains of eclipsed conformations in (I) and (II) the cis olefin is set back from a position directly above the diene by the torsion angles of approximately 60° for C(10)—C(11)—C(12)—C(13) and C(4)—C(5)—C(6)—C(7). These gauche arrangements then connect the planar systems.

The crystalline macrocycles appear to have no great transannular steric crowding as the methyl groups and esters project outside the ring. Some steric crowding may occur from the C(3)/C(14) and C(4)/C(13) methylene-proton interactions. Pitzer strain is minimized, with the exception of the two gauche interactions, by the considerable degree of staggering present in the three planes. Bäyer strain is not evident.

Tricycles having the trans–syn–cis (T–S–C) relative ring-junction stereochemistry resulted from the transannular Diels–Alder reactions of (I) (Michel, Michel-Dewez & Roughton, 1989) and of (II) (Michel, Michel-Dewez, Roughton, Springer & Hoogsteen, 1989). The T–S–C-tricycle-stereochemistry predictions made for the outcome of the transannular Diels–Alder reactions of (I) and (II) – based on the same theory used to predict the results of a similar model series (Lamothe, Ndibwami & Deslongchamps, 1988b) – have thus been subjected to verification in the form of rigorous macrocycle-olefin-geometry and tricycle-stereochemistry determinations from X-ray data. Support for the predictions and for the synthetic strategy has thus been established by the results of these studies.

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