The nearly perpendicular orientation of the phenyl ring relative to the four-atom cyclohexane plane is virtually identical in both (II) and (II'); the respective dihedral angles are 84·2 (2) and 83·0 (2)°. The analogous angle is 110·0 (1)° for (I). If one considers that the shapes of these three molecules may be defined by three 'wedges' meeting at a common vertex (C1) and represented by a plane through each of the cyclohexane and phenyl rings and a plane through the carboxyl group, the three molecules are nearly identical in terms of the relative orientation of these planes. Owing to the geometrical isomerism, of course, the electronic distribution within two of these 'wedges' will be reversed.

There is an intermolecular hydrogen bond between carboxylic acid groups related by the crystallographic inversion center in the structure of (I). The associated metrical parameters are $O1\cdots O2 = 2\cdot631\,(3)$, $HO1\cdots O2 = 1\cdot84\,(3)$ Å with an angle at hydrogen of 172 (3)°. In (II) the intermolecular hydrogen bonding also involves carboxylic dimers formed between head-to-tail arranged independent molecules. The associated geometrical parameters are $O1\cdots O2' = 2\cdot640\,(6)$, $HO1\cdots O2' = 1\cdot54$ Å with an angle at H of 166° and $O1'\cdots O2 = 2\cdot634\,(6)$, $HO1'\cdots O2 = 1\cdot53$ Å with an angle at H of 159°.

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The Structures of Three Strained Cage Molecules

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Abstract. (1) Ethyl {2-hydroxy-9-methyl-6-oxopentacyclo[5.5.0.0^{4,11}.0^{5,9}.0^{8,12}]dodec-2-en-3-yl}carboxylate, $C_{16}H_{18}O_4$, $M_r = 274\cdot32$, monoclinic, $P2_1/n$, $a = 12\cdot564\,(1)$, $b = 6\cdot825\,(1)$, $c = 16\cdot088\,(2)$ Å, $\beta = 101\cdot38\,(1)°$, $V = 1352\cdot4\,(2)$ Å^3, $Z = 4$, $D_x = 1\cdot347$ g cm^{-3}, λ(Mo Kα) = 0·71073 Å, $\mu = 0\cdot90$ cm^{-1}, $F(000) = 584$, $T = 294$ K, $R = 0\cdot0407$ for 1413 reflections. (2) Ethyl {2-hydroxy-1,7-dimethyl-6-oxopentacyclo[5.5.0.0^{4,11}.0^{5,9}.0^{8,12}]dodec-2-en-3-yl}carboxylate, $C_{17}H_{20}O_4$, $M_r = 288\cdot35$, monoclinic, $P2_1/n$, $a = $

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8·167 (1), $b = 14\cdot746$ (2), $c = 12\cdot679$ (2) Å, $\beta = 107\cdot12°$, $V = 1459\cdot4$ (3) Å^3, $Z = 4$, $D_x = 1\cdot312$ g cm^{-3}, λ(Mo $K\alpha$), $\mu = 0\cdot87$ cm^{-1}, $F(000) = 616$, $T = 294$ K, $R = 0\cdot0407$ for 1690 reflections. (3) Dimethyl {6-hydroxy-3-oxo-7-oxatetracyclo[7.3.0.0^{4,12}.-0^{5,10}]dodec-6-yl}methylphosphonate, $C_{14}H_{21}O_6P$, $M_r = 316\cdot29$, triclinic, $P\bar{1}$, $a = 7\cdot608$ (1), $b = 8\cdot994$ (1), $c = 11\cdot595$ (2) Å, $\alpha = 70\cdot11$ (1), $\beta = 70\cdot77$ (1), $\gamma = 84\cdot39$ (1)°, $V = 704\cdot3$ (2) Å^3, $Z = 2$, $D_x = 1\cdot491$ g cm^{-3}, λ(Mo $K\alpha$), $\mu = 2\cdot13$ cm^{-1}, $F(000) = 336$, $T = 293$, $R = 0\cdot0351$ for 2270 reflections. Cage compounds (1) and (2) contain a four-membered ring, three five-membered rings and a six-membered cyclohexene ring. The four-membered rings are slightly puckered, the five-membered rings are in envelope conformations and the six-membered ring is in a half-chair conformation. Molecular mechanics calculations indicate the ring system of compound (1) is 9·6 kJ mol^{-1} less strained than that of (2). Compound (3) lacks the four-membered ring and the six-membered heterocyclic ring is in a chair conformation. The ring system in compound (3) is about 25 kJ mol^{-1} less strained than that in (1).

Introduction. As part of a program to explore the synthesis, chemistry and energetics of novel, substituted cage compounds (Marchand, 1988), we have prepared compounds (1), (2) and (3). The synthesis and structures of several compounds related to (1) and (2) have been reported (Marchand, Arney, Gilardi & Flippen-Anderson, 1987; Marchand, Annapurna, Watson & Nagl, 1988). Compound (3) is included as a reference structure since the strain induced by the four-membered ring has been removed, and any unusual bond length variations related to this strain are absent.

(1)   (2)   (3)

$R = -COOEt$ $R' = -CH_2PO(OMe)_2$
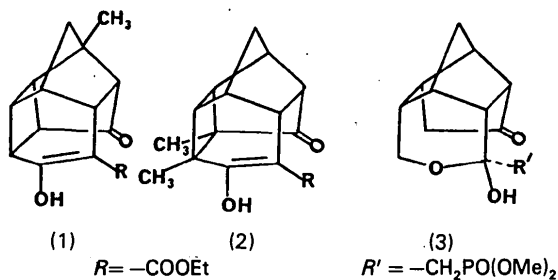
Experimental. Compounds (1) and (2) were synthesized by the boron trifluoride-promoted reaction of the appropriate pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane-8,-11-dione with ethyl diazoacetate. Compound (1), m.p. 379·5–380·0 K, was obtained in 21% yield while compound (2), m.p. 384·5–385·5 K, was prepared in 74% yield. Compound (3) was prepared from the same starting material through formation of a monolactone. Dimethyl methylphosphonate was reacted with n-butyllithium in dry tetrahydrofuran and the mono-

Table 1. *Data collection and refinement parameters for compounds* (1), (2) *and* (3)

	(1)	(2)	(3)
Crystal size (mm)	0·13 × 0·33 × 0·45	0·38 × 0·38 × 0·40	0·30 × 0·40 × 0·50
Unit-cell reflections	25·03–34·87°	26·03–27·90°	48·69–49·99°
2θ range	3–45°	3–45°	3–50°
h,k,l range	±13;0,7;0,17	±8;0,15;0,13	−8,9;−9,10;0,13
Standard reflections	602 234	414 270	162 214
Total intensities	1775	1897	2475
$I \geq 3\sigma(I)$	1413	1690	2270
Transmission factors	0·785–0·839	0·824–0·837	0·833–0·932
Parameters	254	270	274
R	0·0407	0·0407	0·0351
wR	0·0397	0·0596	0·0503
R(all)	0·0554	0·0474	0·0397
wR(all)	0·0413	0·0785	0·0573
S	1·358	1·074	1·840
$(\Delta/\sigma)_{max}$	0·010	0·014	0·007
Max. $\Delta\rho$ (e Å^{-3})	0·15	0·18	0·21
Min. $\Delta\rho$ (e Å^{-3})	−0·14	−0·24	−0·30
g^*	0·00023	0·00262	0·00048
x†	0·00202	—	—

$* w = [\sigma^2(F_o) + gF_o^2]^{-1}$.
$† F = F_c/[1\cdot0 + 0\cdot002xF_c^2/\sin(2\theta)]^{0\cdot25}$.

lactone added. Compound (3), m.p. 403–404 K, was obtained in 32% yield. Single crystals of (1) and (2) were obtained by slow recrystallization from hexane while (3) was recrystallized from an ethyl acetate–hexane mixture. Details of the syntheses are given in the supplementary material. Data were collected on a Nicolet $R3M/\mu$ update of a $P2_1$ diffractometer using the ω scan mode, a variable scan rate (4 to 29·3° min^{-1}) and graphite-monochromated Mo $K\alpha$ radiation. The unit-cell dimensions were obtained by a least-squares refinement of 25 reflections, and space group assignments are consistent with systematic absences and Laue symmetry. Lorentz and polarization corrections were applied and a ψ-scan-based empirical absorption correction was made. The structures were solved by direct methods and refined by a block-cascade least-squares procedure. H atoms were located in difference maps and were refined isotropically. The function minimized was $\sum w(|F_o| - |F_c|)^2$. All computer programs were supplied by Nicolet for Desktop 30 Microeclipse and Nova 4/C configuration (Nicolet Instrument Corporation, 1986), and atomic scattering factors and anomalous-dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974). Table 1 contains crystal and refinement data for compounds (1), (2) and (3) while Tables 2, 3 and 4 give the atomic positional parameters and U_{ij} values for the three compounds. Table 5 presents selected bond distances and Table 6 selected valence angles. Figs. 1, 2 and 3 are drawings of the three molecules.*

Table 2. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($\text{Å}^2 \times 10^3$) *for compound* (1)

	x	y	z	U^*
C(1)	1737 (2)	4102 (4)	6387 (1)	47 (1)
C(2)	1328 (2)	5712 (4)	5797 (1)	43 (1)
O(2)	527 (1)	6808 (3)	5992 (1)	62 (1)
C(3)	1775 (2)	5967 (3)	5106 (1)	39 (1)
C(4)	2658 (2)	4560 (3)	5000 (1)	42 (1)
C(5)	3758 (2)	4689 (3)	5670 (1)	40 (1)
C(6)	3570 (2)	5691 (3)	6456 (1)	41 (1)
O(6)	3875 (1)	7302 (2)	6701 (1)	62 (1)
C(7)	2946 (2)	4287 (3)	6888 (1)	43 (1)
C(8)	3208 (2)	2275 (3)	6553 (1)	43 (1)
C(9)	3984 (2)	2539 (3)	5936 (1)	42 (1)
C(10)	3406 (2)	1358 (4)	5173 (1)	55 (1)
C(11)	2324 (2)	2421 (3)	5090 (1)	48 (1)
C(12)	2071 (2)	2205 (3)	5975 (1)	48 (1)
C(13)	1324 (2)	7404 (4)	4482 (1)	43 (1)
O(13)	585 (1)	8523 (3)	4551 (1)	57 (1)
O(14)	1774 (1)	7416 (2)	3794 (1)	52 (1)
C(14)	1304 (3)	8735 (5)	3115 (2)	66 (1)
C(15)	1749 (2)	8194 (4)	2363 (2)	69 (1)
C(16)	5156 (2)	2045 (4)	6304 (2)	59 (1)

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

Table 3. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($\text{Å}^2 \times 10^3$) *for compound* (2)

	x	y	z	U^*
C(1)	3197 (2)	2873 (1)	7889 (1)	35 (1)
C(2)	2274 (2)	1992 (1)	7835 (1)	36 (1)
O(2)	2749 (2)	1323 (1)	7268 (1)	50 (1)
C(3)	1051 (2)	1900 (1)	8350 (1)	37 (1)
C(4)	658 (2)	2718 (1)	8939 (2)	40 (1)
C(5)	2121 (2)	3020 (1)	10004 (1)	42 (1)
C(6)	3792 (2)	2636 (1)	9966 (1)	40 (1)
O(6)	4584 (2)	2024 (1)	10528 (1)	60 (1)
C(7)	4365 (2)	3150 (1)	9106 (1)	37 (1)
C(8)	3352 (2)	4047 (1)	9007 (1)	37 (1)
C(9)	2264 (2)	4043 (1)	9813 (1)	43 (1)
C(10)	500 (2)	4324 (1)	9060 (2)	51 (1)
C(11)	415 (2)	3573 (1)	8221 (2)	42 (1)
C(12)	2063 (2)	3721 (1)	7909 (1)	38 (1)
C(13)	127 (2)	1054 (1)	8260 (2)	44 (1)
O(13)	395 (2)	394 (1)	7740 (1)	61 (1)
O(14)	−1064 (2)	1033 (1)	8780 (1)	56 (1)
C(14)	−2029 (3)	188 (2)	8707 (2)	70 (1)
C(15)	−3266 (3)	301 (2)	9327 (2)	82 (1)
C(16)	4140 (3)	2949 (1)	7021 (2)	50 (1)
C(17)	6281 (2)	3163 (1)	9322 (2)	53 (1)

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

Table 4. *Atomic coordinates* ($\times 10^4$) *and isotropic thermal parameters* ($\text{Å}^2 \times 10^3$) *for compound* (3)

	x	y	z	U^*
C(1)	1590 (2)	6314 (2)	3271 (2)	32 (1)
C(2)	2166 (2)	7234 (2)	3973 (1)	33 (1)
O(3)	4121 (1)	7677 (1)	3451 (1)	29 (1)
C(4)	4706 (2)	8500 (2)	2104 (1)	28 (1)
C(5)	4292 (2)	7509 (2)	1382 (1)	28 (1)
C(6)	2181 (2)	7186 (2)	1807 (2)	32 (1)
C(7)	2127 (2)	5892 (2)	1243 (2)	38 (1)
C(8)	3382 (2)	4783 (2)	1926 (2)	32 (1)
C(9)	5119 (2)	5793 (2)	1617 (1)	29 (1)
C(10)	5706 (2)	5081 (2)	2822 (2)	31 (1)
O(2)	7285 (2)	5079 (1)	2854 (1)	44 (1)
C(11)	4054 (2)	4235 (2)	3932 (2)	36 (1)
C(12)	2456 (2)	4636 (2)	3367 (1)	32 (1)
O(1)	3695 (2)	9905 (1)	1807 (1)	38 (1)
C(13)	6796 (2)	8858 (2)	1701 (1)	30 (1)
P	7339 (1)	10300 (1)	2290 (1)	31 (1)
O(4)	9281 (2)	10967 (1)	1273 (1)	54 (1)
C(14)	10133 (3)	12322 (2)	1252 (2)	52 (1)
O(5)	7721 (2)	9335 (1)	3588 (1)	53 (1)
C(15)	7669 (3)	10058 (3)	4525 (2)	60 (1)
O(6)	5929 (2)	11524 (1)	2420 (1)	43 (1)

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

Table 5. *Selected bond distances for compounds* (1), (2), (3), *and the averages of three reference structures*

	(1)	(2)	Ref.*	(3)†	
C(1)—C(2)	1·476 (3)	1·493 (2)	1·459 (9)	1·448 (2)	C(2)—O(3)†
C(1)—C(7)	1·579 (3)	1·610 (2)	1·587 (5)	—	
C(1)—C(12)	1·549 (3)	1·561 (2)	1·547 (4)	1·518 (3)	
C(2)—O(2)	1·340 (3)	1·342 (2)	1·345 (3)	—	
C(2)—C(3)	1·352 (3)	1·351 (3)	1·344 (6)	1·416 (2)	O(3)—C(4)
C(3)—C(4)	1·503 (3)	1·501 (3)	1·504 (4)	1·526 (3)	
C(3)—C(13)	1·437 (3)	1·446 (2)	1·448 (1)	1·536 (2)	
C(4)—C(5)	1·578 (3)	1·581 (2)	1·580 (1)	1·578 (2)	
C(4)—C(11)	1·534 (3)	1·533 (2)	1·540 (8)	1·542 (2)	
C(5)—C(6)	1·496 (3)	1·492 (3)	1·493 (8)	1·519 (2)	
C(5)—C(9)	1·539 (3)	1·537 (2)	1·540 (6)	1·550 (2)	
C(6)—O(6)	1·205 (3)	1·213 (2)	1·207 (2)	1·214 (2)	
C(6)—C(7)	1·494 (3)	1·510 (3)	1·522 (5)	1·507 (2)	
C(7)—C(8)	1·535 (3)	1·545 (2)	1·543 (5)	1·521 (3)	
C(7)—C(*exo*)	—	1·508 (3)	1·499 (7)	—	
C(8)—C(9)	1·533 (3)	1·540 (3)	1·539 (5)	1·549 (2)	
C(8)—C(12)	1·543 (3)	1·554 (2)	1·543 (5)	1·569 (2)	
C(9)—C(10)	1·528 (3)	1·533 (2)	1·520 (4)	1·519 (2)	
C(10)—C(11)	1·524 (3)	1·522 (3)	1·515 (9)	1·526 (3)	
C(11)—C(12)	1·526 (3)	1·526 (3)	1·513 (9)	1·536 (2)	
C(1)—C(*exo*)	—	1·523 (3)	—	—	
C(9)—C(*exo*)	1·513 (3)	—	—	—	

* The average of three C(7)-substituted cage compounds (Marchand, Annapurna, Watson & Nagl, 1988). The C(7) substituents are methyl, phenyl and *p*-cyanophenyl.

† Corresponds to numbering of compounds (1) and (2) for comparison purposes but does not correspond to numbering of (3) in drawings or tables. C(2) of (1) and (2) corresponds to O(3) in (3).

Discussion. Cage compounds (1) and (2) and the three related C(7)-substituted compounds (Marchand, Anna-purna, Watson & Nagl, 1988) contain a four-membered ring, three five-membered rings and a six-membered ring giving a cage structure with one open side. Compound (3) contains three five-membered rings and a six-membered heterocyclic ring which for comparison purposes can be considered as a cage with two open sides. In all compounds two of the five-membered rings are fused in a norbornane-like arrangement.

Compounds (1) and (2) and the reference compounds are superimposable, indicating a common conformation with only small displacements from ideal superposition due to substitution patterns. In general, the four-membered rings are slightly puckered with the six-membered cyclohexene rings adopting half-chair conformations and the opposite five-membered rings, envelope conformations. The side fused to the four-membered ring is the base of the envelope. In general,

Table 6. *Selected valence angles for compounds* (1), (2) *and* (3)

	(1)	(2)	(3)
C(2)–C(1)–C(7)	116·5 (2)	114·5 (1)	—
C(2)–C(1)–C(12)	115·9 (2)	113·8 (1)	113·6 (1)
C(7)–C(1)–C(12)	88·3 (2)	88·2 (1)	—
C(1)–C(2)–C(3)	118·4 (2)	119·9 (2)	113·2 (1)
C(1)–C(2)–O(2)	116·6 (2)	116·2 (2)	—
C(2)–C(3)–C(4)	116·5 (2)	117·2 (2)	110·2 (1)
C(2)–C(3)–C(13)	119·5 (2)	119·4 (2)	106·8 (1)
C(3)–C(4)–C(5)	117·0 (2)	115·7 (1)	117·1 (1)
C(3)–C(4)–C(11)	112·0 (2)	112·0 (2)	110·6 (1)
C(5)–C(4)–C(11)	102·0 (2)	102·0 (1)	102·6 (1)
C(4)–C(5)–C(6)	109·9 (2)	109·5 (1)	116·8 (1)
C(4)–C(5)–C(9)	103·0 (2)	102·4 (1)	101·7 (1)
C(6)–C(5)–C(9)	104·6 (2)	104·7 (2)	103·0 (1)
C(5)–C(6)–C(7)	106·6 (2)	108·3 (1)	108·8 (1)
C(1)–C(7)–C(6)	110·7 (2)	110·0 (1)	—
C(1)–C(7)–C(8)	90·0 (2)	89·3 (1)	—
C(6)–C(7)–C(8)	104·0 (2)	102·5 (1)	103·6 (1)
C(7)–C(8)–C(9)	109·2 (2)	110·0 (1)	104·2 (1)
C(7)–C(8)–C(12)	90·1 (2)	90·8 (1)	116·2 (2)*
C(9)–C(8)–C(12)	104·1 (2)	102·8 (1)	102·1 (1)
C(5)–C(9)–C(8)	100·7 (2)	101·2 (1)	99·3 (1)
C(5)–C(9)–C(10)	104·3 (2)	105·2 (1)	105·4 (1)
C(8)–C(9)–C(10)	101·4 (2)	101·9 (1)	104·2 (1)
C(9)–C(10)–C(11)	95·3 (2)	94·6 (1)	94·7 (2)
C(4)–C(11)–C(10)	101·8 (2)	102·3 (1)	101·6 (1)
C(4)–C(11)–C(12)	107·0 (2)	107·5 (1)	106·0 (1)
C(10)–C(11)–C(12)	102·7 (2)	102·4 (1)	103·5 (1)
C(1)–C(12)–C(8)	90·8 (2)	90·8 (1)	116·8 (1)
C(1)–C(12)–C(11)	116·2 (2)	117·5 (1)	111·4 (1)
C(8)–C(12)–C(11)	102·8 (2)	103·2 (1)	103·0 (1)

* Numbering made to correspond to compounds (1) and (2) and not that in Fig. 3, *i.e.* C(2) in table corresponds to O(3) in the figure.

the four-membered rings have three sides of approximately equal length, average = 1·544 (6) Å, and a fourth which is significantly longer, 1·579 (2) Å for (1) and 1·587 (5) Å for the average of the three reference compounds. In compound (2) this bond is further lengthened to 1·610 (2) Å because of the repulsive interactions between the two substituent methyl groups. In all of these compounds the C(4)–C(5) bond is elongated relative to other bonds in the norbornane-like moiety and spans a range of 1·578 (3) to 1·581 (2) Å. This particular pattern of bond lengths is not reproduced by *MM*2 molecular mechanics calculations (Allinger & Yu, 1980) even with changes in dipole interactions and torsional constants. In all calculations the ester side chains are replaced by —COOH. The force constants in these strained ring systems must be adjusted to accommodate the changes in hybridization or overlap.

In the reference compounds with substituents at C(7) the C(6)–C(7) bond tends to be longer than that in the unsubstituted compound (1). In compound (1) the methyl substituent at C(9) has no noticeable effect on the adjacent bond lengths or angles. In compound (2) the methyl group at C(1) and its interaction with the methyl at C(7) affects all of the bonds around C(1).

In compound (3) the absence of the four-membered ring allows the six-membered heterocyclic ring to adopt a chair conformation. The opposite five-membered ring

remains in an envelope conformation; however, the orientation of the envelope has shifted. The absence of the four-membered ring also permits the two ends of the
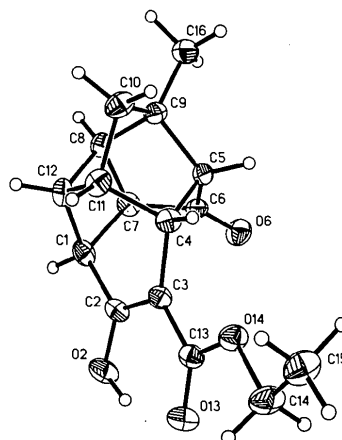

Fig. 1. Drawing of compound (1) with thermal ellipsoids drawn at the 25% probability level.
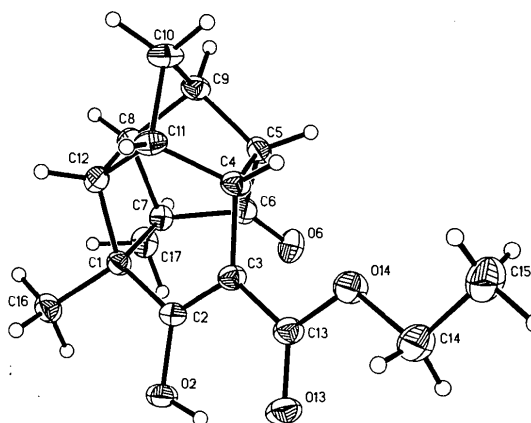

Fig. 2. Drawing of compound (2) with thermal ellipsoids shown at the 25% probability level.
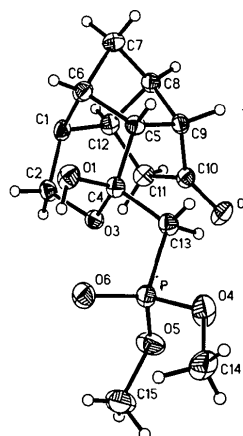

Fig. 3. Drawing of compound (3) with thermal ellipsoids shown at the 35% probability level.

norbornane system to become almost identical in length, $C(1)-C(12) = 1.569$ (2) Å and $C(5)-C(9) = 1.578$ (2) Å (see numbering in Fig. 3). The four external valence angles of the norbornane moiety in (3), e.g. $C(2)-C(1)-C(12)$, open to an average value of 116.7 (5)°. The equivalence of the $C(4)-C(5)$ bonds in (1), (2) and the reference compounds and with the bond in (3) implies the lengthening is not related to strain transmitted by the four-membered ring.

Molecular mechanics calculations predict that compound (3) is the least strained of the compounds discussed, and the formation of the four-membered ring imparts an additional 26·2 and 35·8 kJ mol⁻¹ of strain energy to (1) and (2). The C(7)-substituted compounds have only 16·7 kJ mol⁻¹ more strain energy than (3).

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The Structure of Tris(dimethyldioxolo)triphenylene

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Abstract. Triphenyleno[2,3-*d*:6,7-*d*':10,11-*d*'']tri(2,2-dimethyl-1,3-dioxole), $C_{27}H_{24}O_6$, $M_r = 444.48$, monoclinic, $C2/c$, $a = 22.662$ (8), $b = 9.005$ (1), $c = 24.220$ (5) Å, $\beta = 113.95$ (2)°, $V = 4517$ (2) Å³, $Z = 8$, $D_x = 1.307$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.68$ cm⁻¹, $F(000) = 1872$, $T = 298$ K, final $R = 0.0765$ for 1037 observed unique reflections. Apart from the methyl groups the molecules are planar; the threefold axis perpendicular to the plane of the molecule does not coincide with any of the symmetry operations of the lattice. All intermolecular distances are greater than the sum of van der Waals radii.

Introduction. We report the structure of tris(dimethyldioxolo)triphenylene (TacTPh). The purpose of the investigation has been to gain insight into the packing patterns of peripherally substituted triphenylenes in order to test computer models of the relationship between substitution pattern and molecular organization in organic radical ion crystals (Jørgensen, Bjørnholm & Bechgaard, 1988). Special attention is given to triphenylenes because of their C_3 symmetry which allows degeneracy of the highest occupied molecular orbital. When packed in regular segregated stacks, such molecules constitute potential candidates as donors in radical ion crystals with intriguing magnetic interactions (Miller, Epstein & Reif, 1988).

Experimental. Ketalization of 2,3,6,7,10,11-hexahydroxytriphenylene (Piattelli, Fattorusso, Nicolaus & Magno, 1965) was performed by adding P_2O_5 to a solution of hexahydroxytriphenylene in a 1:1 (v/v) mixture of acetone and acetonitrile followed by reflux for 2 h. Single crystals were obtained by recrystallization from butanol.

A crystal of dimensions $0.4 \times 0.1 \times 0.05$ mm was used for data collection on a CAD-4 diffractometer with graphite-monochromatized Mo $K\alpha$ radiation.