and C19—C20 bond lengths are shorter in β- than in α-amyryl acetate. The two structures are otherwise very similar, although a shorter C12—C13 bond length, 1·316 as against 1·334 Å, and a larger C11—C12—C13—C14 dihedral angle, 4·5 as against 0·3°, are found for β- than for α-amyryl acetate.

A parallel structural determination of α-amyryl acetate was carried out and gave results in good agreement with those previously published (Grynpas & Lindley, 1979).

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syn-8,*syn*-13-Bis(benzoyloxy)heptacyclo[7.6.0.0^{2,7}.0^{4,14}.0^{5,12}.0^{6,10}.0^{11,15}]pentadecan-3-one

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Abstract. $C_{29}H_{24}O_5$, $M_r = 452·52$, monoclinic, $I2/a$ (non-standard setting of $C2/c$), $a = 7·979$ (2), $b = 21·209$ (6), $c = 12·614$ (4) Å, $\beta = 90·49$ (2)°, $V = 2134·7$ (10) Å^3, $Z = 4$, $D_x = 1·408$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0·73$ mm^{-1}, $F(000) = 952$, $T = 295$ K, final $R = 0·068$, $wR = 0·062$ for 1012 observed reflections. The cage system exhibits a great deal of strain shown in both the bond lengths and angles. Of the 20 C—C—C angles in the cage moiety five are significantly larger than normal [ave. = 115·3 (4)°] and 13 are significantly smaller than normal [ave. = 101·8 (4)°]. The internal angle at C(8) at 95·4 (4)° is extremely small. Molecular-mechanics calculations predicted the angle strain but not the unusual bond lengths.

Introduction. In a recent study of the thermal reaction of (pentacarbonyl)iron with 7-(benzoyloxy)norbornadiene, a novel cage ketone, $C_{29}H_{24}O_5$ (1), was isolated as a minor reaction product (0·4% yield; Marchand, Earlywine & Heeg, 1986). Examination of its proton and ^{13}C NMR spectra indicated the absence of olefinic absorptions. In addition, the proton noise-decoupled ^{13}C NMR spectrum of (1) displays only 13 signals, a result which suggests that this compound possesses twofold symmetry. On the basis of spectral information available at that time, structure (1*a*) was assigned tentatively to this compound (Marchand, Earlywine & Heeg, 1986), although it was recognized that IR and NMR spectral information was not sufficient to determine its structure uniquely. It was not

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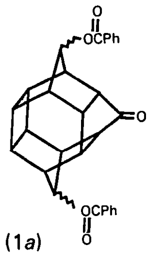
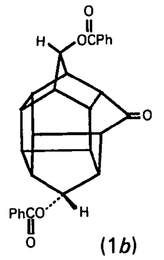
possible to obtain a single crystal of sufficient quality to permit direct determination of the structure of this compound *via* single-crystal X-ray structural analysis. More recently, a good-quality single crystal has been obtained *via* careful fractional recrystallization of this compound from chloroform–hexane mixed solvent. We now report the results of the single-crystal X-ray structural analysis of (1) which showed that the structure is (1*b*).



(1*a*)

[OC(O)Ph groups either both *syn* or both *anti* to C=O group]



(1*b*)

Experimental. Colorless crystal, 0·04 × 0·08 × 0·82 mm, automated Nicolet *R*3*M* diffractometer with incident-beam graphite monochromator, 20 centered reflections with $25 < 2\theta < 80°$ used for determining lattice parameters; $[(\sin\theta)/\lambda]_{max} = 0{\cdot}514$ Å^{-1}, range of *hkl*: $-7 \le h \le 7, 0 \le k \le 21, 0 \le l \le 12$. Standards $3\bar{3}2$, 066, 060, monitored every 60 reflections with random variation of 2·5% over data collection, θ–2θ mode, scan width $(2{\cdot}2 + \Delta 2\theta_{\alpha_1\alpha_2})°$, scan rate a function of count rate (6° min^{-1} minimum, 30° min^{-1} maximum), 4058 reflections measured, 1233 unique, $R_{int} = 0{\cdot}022$, 1012 observed $[F_o > 3\sigma(F_o)]$. Data corrected for Lorentz and polarization effects, absorption ignored.

Structure solved by direct methods. Full-matrix least squares. All programs used part of package provided with Nicolet Micro-VAX system (Sheldrick, 1987). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0{\cdot}00025$. 212 parameters: atom coordinates and anisotropic temperature factors for all non-H atoms, coordinates and isotropic temperature factors for all H. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Final $R = 0{\cdot}068$, $wR = 0{\cdot}062$. Max. $\Delta/\sigma = 0{\cdot}010$, $S = 2{\cdot}23$. Final difference Fourier map excursions 0·38 and $-0{\cdot}25$ e Å^{-3}.*

Discussion. Atom numbering for Tables 1 and 2 follows that shown in Fig. 1. The molecule lies on a crystallographic twofold axis which passes through the O(1)—C(3) bond (Fig. 2). The norbornadiene ring system is highly strained. Most of the C(sp^3)—C(sp^3) bonds are either significantly longer or shorter (Table 2) than values usually found for such bonds in unstrained cages (1·52–1·53 Å) (George & Gilardi, 1983, 1984). The C—C—C angles in the cage system provide further evidence of strain (Table 2). Of the 20 C—C—C angles

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

	x	*y*	*z*	U_{eq}*
O(1)	−2500	5017 (2)	0	73 (2)
O(2)	1147 (4)	6445 (1)	1936 (3)	79 (1)
O(3)	1397 (6)	6991 (2)	3434 (3)	125 (2)
C(1)	−510 (7)	6532 (2)	−194 (4)	67 (2)
C(2)	−1163 (6)	5979 (2)	498 (3)	56 (2)
C(3)	−2500	5596 (3)	0	55 (2)
C(6)	−3259 (7)	6767 (2)	1068 (4)	76 (2)
C(7)	−1868 (6)	6332 (3)	1448 (4)	70 (2)
C(8)	−412 (7)	6780 (3)	1683 (5)	86 (2)
C(9)	−441 (8)	7086 (2)	641 (5)	85 (2)
C(10)	−2181 (9)	7323 (3)	533 (5)	100 (3)
C(16)	1888 (7)	6576 (2)	2855 (4)	73 (2)
C(17)	3306 (5)	6163 (2)	3094 (3)	55 (2)
C(18)	4117 (7)	6245 (2)	4059 (4)	66 (2)
C(19)	5360 (7)	5841 (3)	4362 (5)	78 (2)
C(20)	5868 (7)	5359 (3)	3715 (5)	80 (2)
C(21)	5074 (7)	5279 (3)	2752 (5)	76 (2)
C(22)	3808 (6)	5673 (2)	2438 (4)	63 (2)

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

Table 2. *Selected bond lengths* (Å) *and angles* (°): *comparison of X-ray and MM2 predicted values*

	X-ray	*MM2*		X-ray	*MM2*
O(1)—C(3)	1·229 (6)	1·209	C(6)—C(7)	1·517 (6)	1·547
O(2)—C(8)	1·466 (6)	1·408	C(6)—C(10)	1·611 (8)	1·546
C(3)—C(2)*	1·476 (6)	1·501	C(7)—C(8)	1·527 (7)	1·537
C(1)—C(2)	1·555 (6)	1·545	C(8)—C(9)	1·465 (7)	1·531
C(1)—C(9)	1·579 (6)	1·550	C(9)—C(10)	1·482 (8)	1·533
C(1)—C(6a)	1·552 (7)	1·547	C(10)—C(10a)	1·435 (7)	1·516
C(2)—C(7)	1·525 (6)	1·536			
C(2)—C(3)—O(1)	123·3 (4)		C(6)—C(7)—C(2)	108·9 (4)	
C(2)—C(3)—C(2a)	113·4 (5)		C(8)—C(7)—C(2)	100·0 (4)	101·0
C(9)—C(1)—C(6a)	101·3 (5)	105·3	C(7)—C(8)—C(9)	95·4 (4)	92·8
C(9)—C(1)—C(2)	101·3 (4)	101·1	C(7)—C(8)—O(2)	112·6 (4)	
C(6a)—C(1)—C(2)	115·3 (5)	116·5	C(9)—C(8)—O(2)	114·6 (5)	
C(1)—C(2)—C(7)	101·4 (4)	102·3	C(1)—C(9)—C(8)	105·7 (4)	
C(1)—C(2)—C(3)	114·8 (4)		C(1)—C(9)—C(10)	99·5 (4)	100·8
C(7)—C(2)—C(3)	109·5 (4)		C(8)—C(9)—C(10)	103·9 (6)	100·4
C(7)—C(6)—C(10)	100·7 (4)	101·7	C(9)—C(10)—C(10a)	114·3 (5)	
C(7)—C(6)—C(1a)	119·1 (5)	118·1	C(9)—C(10)—C(6)	102·4 (4)	103·4
C(10)—C(6)—C(1a)	105·9 (5)		C(1a)—C(10)—C(6)	101·9 (5)	104·9
C(6)—C(7)—C(8)	103·7 (5)	103·1			

* sp^3–sp^2 bond; all other C—C bonds are sp^3–sp^3. The 'target value' for all the sp^3–sp^3 cage bonds for the *MM*2 calculations was 1·523 Å.

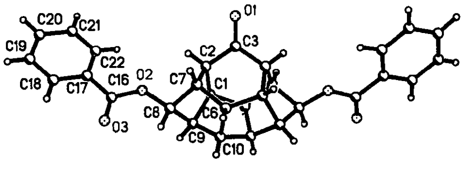


Fig. 1. A perspective view showing the results of the X-ray study on (1).

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and full listings of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44243 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

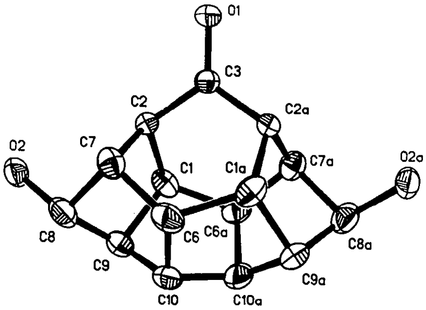

Fig. 2. A view of (1) showing the twofold axis. The benzoyl groups, not shown, are also related by this twofold axis. The thermal ellipsoids are shown at the 20% probability level.

in the norbornadiene moiety five are significantly larger than normal tetrahedral angles [ave. = 115·3 (4)°] and 13 are significantly smaller than the expected value of 109·5° [ave. = 101·8 (4)°]. The internal angle at the bridge atom, C(8), is extremely small [C(7)—C(8)—C(9) = 95·4 (4)°]. Molecular-mechanics calculations were carried out with the 1980 version of program *MM*2 (Allinger, 1976) to see if the strain found in this system could be mathematically predicted. In fact, all the distances along the C(8)—C(9)—C(10)—C(10a)—C(9a)—C(8a) chain are extraordinarily short [ave. = 1·466 (7) Å] yet are predicted to be 1·52 or 1·53 Å by *MM*2. An analysis of the thermal ellipsoids for these atoms indicated corrections of only 0·007 Å for these bonds, indicating that the shortening is not due to thermal motion. These bonds involve atoms separated from double-bonding systems by at least two single bonds. Therefore, while these calculations did predict the angular strain, no meaningful correlations were noted between the bond lengths found in the X-ray study and those calculated by the molecular-mechanics program (Table 2).

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Structure of an Antileukaemic Agent, 5,5′,10,10′-Dihydro-5,5′-dioxo-1,1′,3,3′-tetraphenyl-10,10′-bi(benz[*g*])isoquinoline*

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Abstract. $C_{50}H_{32}N_2O_2$, $M_r = 692{\cdot}78$, triclinic, $P\bar{1}$, $a = 10{\cdot}4411\ (9)$, $b = 11{\cdot}746\ (1)$, $c = 15{\cdot}929\ (1)$ Å, $\alpha = 86{\cdot}21\ (1)$, $\beta = 82{\cdot}63\ (1)$, $\gamma = 64{\cdot}57\ (1)°$, $V = 1750{\cdot}3$ Å^3, $Z = 2$, $D_m = 1{\cdot}322\ (2)$, $D_x = 1{\cdot}315$ g cm^{-3}, Cu $K\alpha$ ($\lambda = 1{\cdot}5418$ Å), $\mu = 5{\cdot}44$ cm^{-1}, $F(000) = 724$, $T = 293$ K, $R = 0{\cdot}041$ for 3636 unique reflections with $F > 5{\cdot}0\sigma(F)$. The molecule consists of two similar fragments linked through a weak $C(sp^3)$—$C(sp^3)$ bond of length 1·617 (6) Å. The nitrogens of the pyridine rings are not protonated. The rotations of the phenyl rings from the plane of the pyridine ring prevent the conjugation of the unsaturated systems. The molecular packing in the unit cell is stabilized by van der Waals forces.

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