$(C_6F_5)_3PF_2$ (Sheldrick, 1975). Both phosphoranes crystallize in space group *Pbcn*, with the molecule lying on a crystallographic diad axis. However, whereas the perfluoro derivative displays a regular propeller-like arrangement of the three phenyl rings, one of the phenyl rings in $(C_6H_5)_3PF_2$ opposes the propeller arrangement of the other two.

The P—F bond length in $(C_6H_5)_3PF_2$, at 1.663 (2) Å, is slightly longer than that in $(C_6F_5)_3PF_2$ [1.636 (2) Å], perhaps as a result of the increased electrophilicity of P in $(C_6F_5)_3PF_2$ owing to increased electron withdrawal by the perfluoroaryl substituents. The aryl rings in $(C_6F_5)_3PF_2$ display smaller dihedral angles with the equatorial plane (*ca* 33.8, 37.9 and 37.9°) than the phenyl rings in $(C_6H_5)_3PF_2$ (*ca* 51.5, 63.4 and 63.4°). Theoretical studies (Hoffmann, Howell & Muetterties, 1972), suggest that π donors will align their orbitals parallel to the equatorial plane of pentavalent phosphorus, while π acceptors prefer to align their orbitals perpendicular to this plane. The larger dihedral angles observed for $(C_6H_5)_3PF_2$, bearing better π donor substituents, suggest a shift in structure toward the predicted conformation. However, the P—C bond lengths, at approximately 1.82 Å, are not statistically different in the two phosphoranes, suggesting that π-bonding effects may be quite subtle.

Other metrical parameters in the two phosphoranes are nearly identical and in accord with near-ideal trigonal bipyramidal geometry: the F—P—F bond angle is 178.3 (2)° in $(C_6H_5)_3PF_2$ and 180.0° in $(C_6F_5)_3PF_2$, and the C—P—C angles in both are approximately 120°.

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Three Substituted Hexahydro-5,8-dimethanonaphthalenes

BY WILLIAM H. WATSON,* RAM P. KASHYAP AND MARIUSZ KRAWIEC

Department of Chemistry, Texas Christian University, Fort Worth, Texas 76129, USA

AND ALAN P. MARCHAND* AND SHAO-PO LU

Department of Chemistry, University of North Texas, PO Box 5068, Denton, Texas 76203, USA

Abstract. rel-(4a*S*,5*S*,8*S*,8a*S*)-1,4,4a,5,8,8a-Hexahydro-2-methoxy-5-methyl-5,8-methanonaphthalene-5,8-dione (2*a*), $C_{13}H_{14}O_3$, $M_r = 218.28$, monoclinic, $P2_1/c$, $a = 13.561$ (4), $b = 6.598$ (1), $c = 14.108$ (4) Å, $\beta = 118.37$ (2)°, $V = 1110.8$ (6) Å^3, $Z = 4$, $D_x = 1.310$ g cm^{-3}, λ(Mo *K*α) = 0.71073 Å, $\mu = 0.86$ cm^{-1}, $F(000) = 464$, $T = 298$ K, $R = 0.0969$ (*wR* = 0.0422) for 1014 reflections. rel-(4a*S*,5*S*,8*S*,8a*S*)-2,4a-Dibromo-1,4,4a,5,8,8a-hexahydro-5-methyl-5,8-methanonaphthalene-5,8-dione (2*b*), $C_{12}H_{10}Br_2O_2$, $M_r = 346.05$, monoclinic, $P2_1/n$, $a = 6.556$ (2), $b = 25.739$ (10), $c = 7.436$ (5) Å, $\beta = 111.25$ (4)°, $V = 1170$ (1) Å^3, $Z = 4$, $D_x = 1.970$ g cm^{-3}, λ(Mo *K*α) = 0.71073 Å, $\mu = 68.46$ cm^{-1}, $F(000) = 672$, $T = 298$ K, $R = 0.0674$ (*wR* = 0.0455) for 1389 observed reflections. rel-(4*R*,4a*R*,5*R*,8*S*,9*S*)-1,4,4a,5,8,8a-Hexa-

* To whom correspondence should be addressed

hydro-4-hydroxy-3-methoxy-8-methyl-5,8-methano-naphthalen-1-one (6a), $C_{13}H_{16}O_3$, $M_r = 220.27$, monoclinic, Cc, $a = 10.827$ (4), $b = 8.395$ (2), $c = 12.997$ (2) Å, $\beta = 105.34$ (2)°, $V = 1139.2$ (4) Å^3, $Z = 4$, $D_x = 1.280$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 1.00$ cm^{-1}, $F(000) = 472$, $T = 298$ K, $R = 0.0352$ ($wR = 0.0480$) for 1268 observed reflections. The three compounds contain a six-membered ring cis-fused to the end of a norbornene moiety. The atoms of the norbornene double bond exhibit the normal pyramidalization observed for this class of compounds. One C—C bond adjacent to the methyl-substituted bridge averages 1.587 (3) Å.

Introduction. Recently, we have been involved in a study of the synthesis and chemistry of substituted hexahydro-1,4-methanonaphthalenes. These compounds are of interest as synthetic precursors to pentacyclic cage compounds (Marchand, LaRoe, Sharma, Suri & Reddy, 1986; Marchand, 1989) which, in turn, serve as intermediates in the synthesis of polyquinone natural products (Mehta, Srikrishna, Reddy & Nair, 1981; Marchand, 1991). As part of this program, the Diels–Alder cycloadditions of a 60:40 mixture of 1-methyl- and 2-methylcyclopentadienes (Csicsery, 1960) with 2-methoxy-*p*-benzoquinone (1a) and with 2,5-dibromo-*p*-benzoquinone (1b) were investigated individually. Each of these reactions is expected to produce a mixture of as many as four isomeric *endo* [4 + 2] cycloadducts [(2a)–(5a) and (2b)–(5b), respectively].

In each case, a single pure isomer was isolated *via* careful fractional recrystallization of the product mixture. Simple integration of the proton NMR spectrum of each of the materials thereby obtained revealed the presence of three vinyl protons. This observation requires that the isomer, m.p. 416–417 K, obtained *via* fractional recrystallization (from ethyl acetate–hexane) of the mixture of cyclo-adducts produced *via* Diels–Alder reaction of (1a) with methylcyclopentadienes must possess either structure (2a) or (5a). Similarly, the single pure isomer, m.p. 369–370 K, obtained *via* fractional recrystallization (from hexane) of the product mixture that resulted *via* Diels–Alder cycloaddition of methylcyclopentadienes with (1b) must possess either structure (2b) or (5b). In each case, unequivocal structural assignment was made by single-crystal X-ray analysis. Thus, the structures of the cyclo-adducts obtained *via* fractional recrystallization of the mixture of isomeric Diels–Alder cycloadducts were shown to be (2a) and (2b), respectively.

It was of further interest to study the reduction of (2a) with excess sodium hydroborate in the presence of cerium(III) chloride heptahydrate (Marchand, LaRoe, Sharma, Suri & Reddy, 1986). Under the conditions employed in this reaction (*i.e.* reaction run in methanol solvent at room temperature for 18 h), only one of the two ketone carbonyl groups in (2a) was reduced. Furthermore, this reduction occurred stereospecifically to afford a single pure product, m.p. 401-402 K, in high yield.

(1a) X=OCH₃; Y=H
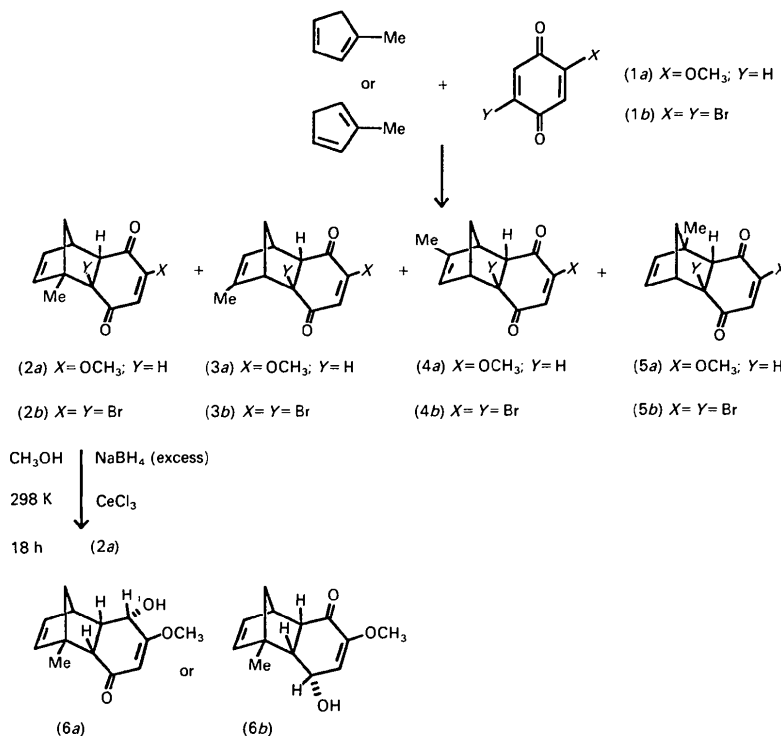
(1b) X=Y=Br

(2a) X=OCH₃; Y=H   (3a) X=OCH₃; Y=H   (4a) X=OCH₃; Y=H   (5a) X=OCH₃; Y=H

(2b) X=Y=Br   (3b) X=Y=Br   (4b) X=Y=Br   (5b) X=Y=Br

CH₃OH | NaBH₄ (excess)

298 K | CeCl₃

18 h   (2a)

(6a)   (6b)

Table 1. *Crystal, data collection and refinement parameters*

	(2a)	(2b)	(6a)
Crystal data			
Crystal size (mm)	0.50 × 0.15 × 0.05	0.40 × 0.25 × 0.23	0.60 × 0.28 × 0.25
Color	Colorless	Colorless	Colorless
2θ range (°) for lattice parameters	6.01–15.09	23.62–28.98	23.34–28.56
Systematic absences	$h0l, l = 2n + 1$	$h0l, h + l = 2n + 1$	$hkl, h + k = 2n + 1$
	$0k0, k = 2n + 1$	$0k0, k = 2n + 1$	$h0l, h, l = 2n + 1$
Data collection			
2θ range (°)	3–50	3–50	3–55
hkl	−16, 16; 0, 7;	−7, 7; 0, 30;	−14, 14; 0, 10;
	0,16	0,8	0,16
Monitored reflections	$\overline{2}01, 1\overline{1}\overline{3}$	$\overline{2}21, \overline{2}\overline{3}1$	$\overline{2}0\overline{6}, \overline{1}\overline{3}0$
Total reflections	2231	2311	2920
Independent reflections	1921	2062	1367
R_{int}	0.024	0.029	0.051
Reflections $I > 3\sigma(I)$	1014	1389	1268
Transmission factors	Not applied	0.228–0.169	0.970–0.911
Refinement			
R (R all data)	0.0969 (0.172)	0.0674 (0.105)	0.0352 (0.0398)
wR (wR all data)	0.0422 (0.0466)	0.0455 (0.0480)	0.0480 (0.0482)
Number of parameters	171	164	184
S	1.927	1.778	1.466
$(\Delta/\sigma)_{max}$	0.004	0.012	0.016
Largest peaks in final difference map (e Å^{-3})	−0.39; 0.37	−0.67; 0.71	−0.13; 0.16
g (weighting scheme)	0.0000	0.0008	0.0059
x (× 10^6)*	1.76 (9)	2.08 (8)	3.76 (9)

$$*F = F_c/[1.0 - xF_c^2/\sin(2\theta)]^{0.25}.$$

Since the corresponding reduction of substituted 1α,4α,4aα,5α,8β,8aα-hexahydro-1,4-methanonaphthalene-5,8-diones has been shown previously to proceed stereospecifically to afford the corresponding 5β,8β-diols (Marchand, LaRoe, Sharma, Suri & Reddy, 1986; Flippen-Anderson, Gilardi, George, Marchand & Jin, 1987), it seems reasonable to expect that NaBH$_4$–CeCl$_3$-promoted reduction of (2a) might proceed with similar stereochemical consequences, thereby affording either ketol (6a) or (6b). Application of X-ray crystallographic methods resolved this ambiguity; the structure of this ketol was demonstrated unequivocally to be (6a).

Experimental. All data were collected on a Nicolet R3M/μ update of a $P2_1$ diffractometer using the ω-scan mode with a variable scan rate of 4 to 29.3° min^{-1} and graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å). The data were corrected for Lorentz–polarization effects, and a ψ-scan absorption correction was applied. Absence of suitable reflections precluded a ψ-scan correction for (2a). Lattice parameters were obtained from a least-squares refinement of 25 reflections, 15 for (2a). The structures were solved by direct methods and refined by a block-cascade least-squares technique. All H atoms were located in difference maps; however, methyl and methylene H atoms were allowed to ride on the attached atoms. The function minimized was $\sum w(|F_o| - |F_c|)^2$ with $w = [\sigma^2(F_o) + gF_o^2]^{-1}$. All computer programs supplied by Nicolet for Desktop

30 Microeclipse and Nova 4/C configuration (Sheldrick, 1985). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Crystal, data collection and refinement parameters are given in Table 1. Tables 2, 3, and 4 give the atomic positional parameters for compounds (2a), (2b) and (6a), respectively, while Tables 5 and 6 give selected bond lengths and angles. Figs. 1, 2, and 3 are thermal ellipsoid drawings of the three compounds.*

Discussion. The small crystal of (2a) with Mo radiation gave a number of weak reflections leading to a relatively large R value; however, the weighted R was the smallest of the three structures and the standard deviations were intermediate between those of (2b) and (6a). The three compounds contain a six-membered ring cis-fused to the end of a norbornene moiety. The six-membered ring in (2a) is distorted slightly from planarity toward a boat conformation with both carbonyl O atoms lying on the same side and out of the plane by 0.35 (1) and 0.36 (1) Å. The six-membered ring in (2b) is more planar with the two carbonyl O atoms lying on opposite sides and

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

	x	y	z	U*
C(1)	8004 (4)	6654 (9)	3449 (4)	43 (3)
C(2)	6880 (4)	6693 (9)	3531 (4)	34 (3)
C(3)	6326 (4)	8699 (8)	3243 (4)	44 (3)
O(3)	6697 (3)	10137 (6)	3862 (3)	69 (2)
C(4)	5337 (5)	9033 (10)	2201 (4)	39 (3)
C(5)	4781 (4)	7439 (10)	1569 (4)	39 (3)
O(5)	3871 (3)	7560 (6)	603 (3)	52 (2)
C(6)	5086 (4)	5317 (10)	1916 (4)	46 (3)
O(6)	4438 (3)	3955 (6)	1435 (4)	87 (3)
C(7)	6224 (4)	4880 (8)	2834 (4)	42 (3)
C(8)	7023 (5)	4057 (9)	2403 (4)	56 (3)
C(9)	7084 (5)	5617 (11)	1680 (4)	64 (3)
C(10)	7656 (4)	7138 (10)	2275 (4)	58 (3)
C(11)	8147 (4)	4340 (9)	3410 (4)	62 (3)
C(12)	8970 (4)	7862 (10)	4300 (4)	72 (3)
C(13)	3498 (4)	9560 (9)	158 (4)	60 (3)

* 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* (6a)

	x	y	z	U*
C(1)	−43 (2)	5985 (3)	−1235 (2)	43 (1)
C(2)	917 (2)	4523 (3)	−1096 (2)	39 (1)
C(3)	227 (2)	2972 (3)	−1117 (2)	44 (1)
O(3)	−486 (2)	2512 (2)	−1970 (2)	65 (1)
C(4)	374 (3)	2083 (3)	−149 (2)	45 (1)
C(5)	1269 (2)	2442 (3)	753 (2)	38 (1)
O(5)	1465	1663 (2)	1674	49 (1)
C(6)	2289 (2)	3694 (3)	825 (2)	37 (1)
O(6)	2607 (2)	4469 (2)	1828 (2)	44 (1)
C(7)	1964 (2)	4947 (3)	−58 (2)	40 (1)
C(8)	1480 (3)	6561 (3)	275 (2)	49 (1)
C(9)	262 (3)	6245 (3)	575 (2)	53 (1)
C(10)	−633 (3)	5889 (3)	−305 (2)	52 (1)
C(11)	938 (3)	7311 (3)	−832 (2)	52 (1)
C(12)	−943 (3)	6196 (4)	−2339 (2)	60 (1)
C(13)	648 (3)	350 (3)	1737 (3)	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* (2b)

	x	y	z	U*
C(1)	3241 (14)	4332 (3)	7532 (12)	40 (4)
C(2)	2539 (13)	3903 (3)	5884 (11)	32 (3)
Br(2)	910 (2)	4221 (1)	3339 (1)	50 (1)
C(3)	4472 (14)	3657 (4)	5586 (13)	45 (4)
O(3)	5827 (11)	3903 (3)	5250 (11)	71 (4)
C(4)	4736 (16)	3094 (4)	5861 (14)	45 (4)
C(5)	3341 (15)	2785 (3)	6271 (12)	40 (4)
Br(5)	3827 (2)	2066 (1)	6562 (2)	58 (1)
C(6)	1413 (14)	2975 (3)	6602 (12)	40 (4)
O(6)	161 (11)	2685 (2)	6972 (11)	69 (4)
C(7)	993 (14)	3550 (3)	6468 (13)	34 (4)
C(8)	1099 (18)	3784 (3)	8428 (15)	47 (4)
C(9)	3463 (19)	3698 (4)	9785 (14)	56 (5)
C(10)	4623 (19)	4015 (4)	9217 (15)	51 (5)
C(11)	1137 (15)	4365 (3)	7975 (14)	47 (4)
C(12)	4203 (18)	4828 (4)	7116 (15)	61 (5)

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

Table 5. *Selected bond lengths* (Å) *for compounds* (2a), (2b) *and* (6a)

	(2a)	(2b)	(6a)
C(1)—C(2)	1.584 (9)	1.590 (11)	1.588 (3)
C(1)—C(10)	1.525 (8)	1.495 (12)	1.511 (4)
C(1)—C(11)	1.543 (8)	1.532 (15)	1.533 (4)
C(1)—C(12)	1.516 (7)	1.503 (14)	1.516 (3)
C(2)—C(3)	1.481 (8)	1.504 (14)	1.498 (3)
C(2)—C(7)	1.536 (7)	1.536 (14)	1.557 (3)
C(3)—C(4)	1.461 (6)	1.465 (13)	1.436 (4)
C(3)—O(3)	1.225 (6)	1.189 (13)	1.233 (3)
C(4)—C(5)	1.353 (8)	1.327 (15)	1.342 (4)
C(5)—C(6)	1.476 (9)	1.457 (14)	1.510 (3)
C(6)—C(7)	1.497 (6)	1.503 (12)	1.528 (3)
C(6)—O(6)	1.214 (7)	1.213 (12)	1.415 (3)
C(7)—C(8)	1.570 (10)	1.555 (15)	1.555 (3)
C(8)—C(9)	1.478 (10)	1.528 (14)	1.494 (5)
C(8)—C(11)	1.523 (6)	1.534 (12)	1.539 (3)
C(9)—C(10)	1.301 (8)	1.287 (18)	1.323 (4)
C(2)—Br(2)	—	1.981 (7)	—
C(5)—Br(5)	—	1.878 (8)	—
C(5)—O(5)	1.337 (5)	—	1.332 (3)
O(5)—C(13)	1.446 (7)	—	1.428 (3)

Table 6. *Selected bond angles* (°) *for compounds* (2a), (2b) *and* (6a)

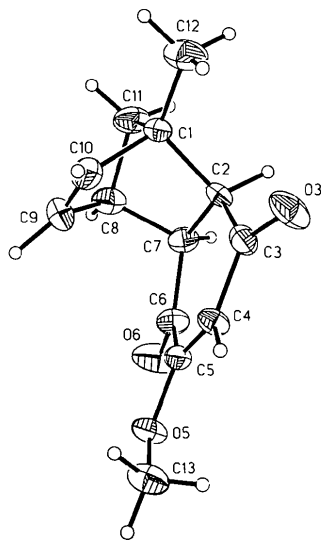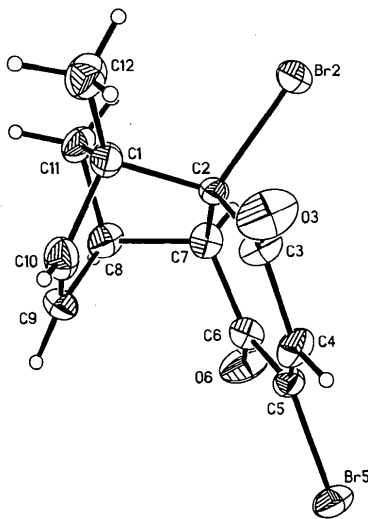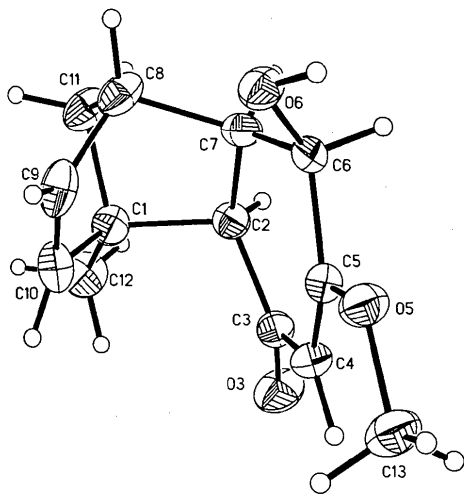|	(2a)	(2b)	(6a)
C(2)—C(1)—C(10)	105.3 (4)	100.6 (7)	105.7 (2)
C(2)—C(1)—C(11)	99.2 (5)	99.9 (6)	98.5 (2)
C(2)—C(1)—C(12)	115.6 (5)	117.2 (8)	115.8 (2)
C(10)—C(1)—C(11)	98.5 (5)	99.8 (8)	99.6 (2)
C(1)—C(2)—C(7)	103.0 (5)	103.2 (7)	103.1 (2)
C(3)—C(2)—C(7)	117.1 (4)	118.0 (7)	117.4 (2)
C(2)—C(3)—C(4)	121.0 (5)	117.4 (9)	119.8 (2)
C(2)—C(3)—O(3)	120.9 (4)	122.7 (8)	118.7 (2)
C(4)—C(3)—O(3)	118.1 (5)	119.8 (9)	121.5 (2)
C(3)—C(4)—C(5)	120.2 (5)	124.6 (10)	122.4 (2)
C(4)—C(5)—C(6)	122.6 (4)	123.4 (8)	123.9 (2)
C(5)—C(6)—C(7)	118.7 (5)	118.0 (8)	114.4 (2)
C(5)—C(6)—O(6)	120.2 (4)	122.1 (8)	111.9 (2)
O(6)—C(6)—C(7)	121.0 (5)	119.9 (9)	109.1 (2)
C(2)—C(7)—C(6)	116.7 (5)	118.5 (8)	117.6 (2)
C(2)—C(7)—C(8)	102.8 (5)	103.1 (7)	102.6 (2)
C(7)—C(8)—C(9)	107.5 (5)	104.7 (9)	107.7 (2)
C(7)—C(8)—C(11)	99.5 (5)	99.8 (8)	99.7 (2)
C(9)—C(8)—C(11)	101.4 (5)	101.1 (8)	99.9 (2)
C(8)—C(9)—C(10)	107.7 (5)	105.7 (9)	108.1 (3)
C(1)—C(10)—C(9)	108.7 (6)	111.7 (10)	108.2 (3)
C(1)—C(11)—C(8)	93.9 (4)	94.7 (8)	94.7 (2)

Fig. 1. Drawing of compound (2a). Thermal ellipsoids are drawn at the 35% probability level. H atoms are represented by spheres of arbitrary size.

out of the plane by only 0.05 (1) Å. The six-membered ring in (6a) is folded along a C(3)···C(6) axis with an angle of 163.2 (7)°. C(1)—C(2) is consistently the longest bond in the three structures with an average value of 1.587 (3) Å. This may be compared with the C(7)—C(8) bond of 1.560 (9) Å. The *AMPAC* program package (version 2.1) (Dewar, Zoebisch, Healy & Stewart, 1985) with *AM*1 parameters gives values of 1.567 and 1.561 Å for the two bonds in (2a) while *MNDO* computes the bonds to be 1.594 and 1.575 Å. The structures of compounds (7) (Flippen-Anderson, Gilardi, George, Marchand & Jin, 1987) and (8) (Watson, Nagl, Kashyap, Marchand & Zhao, 1989) have been reported. In compound (7) the two carbonyl groups have been hydrogenated and the C(1)—C(2) and C(7)—C(8) bonds are 1.551 (5) and 1.545 (5) Å. In compound (8) the C(1) methyl group has been replaced by a methyl at the ring juncture and one on the double bond of the norbornene. The bond with the attached methyl is 1.564 (3) Å while the other is 1.547 (3) Å. The double bonds at C(4)—C(5) and C(9)—C(10) can be closed photochemically to a four-membered ring. The C···C separations between these two bonds range from 3.28 (1) to 3.50 (1) Å.

(7)   (8)
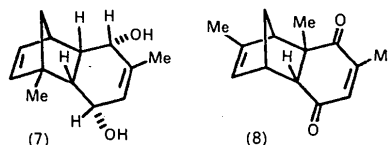
Although the errors in the H positions are large, the pyramidalization is consistent with that observed in other norbornene systems (Watson, 1983; Watson, Kashyap & Plummer, 1991). The angle of bend of the π system (3 to 9°) and the twist (0.1 to 4.0°) and pyramidalization angles (1.5 to 13.8°) (Ermer & Mason, 1982; Ermer, Bell & Mason, 1989) are included in the supplementary material.

Fig. 2. Drawing of compound (2b). Thermal ellipsoids are drawn at the 35% probability level. H atoms are represented by spheres of arbitrary size.

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Fig. 3. Drawing of compound (6a). Thermal ellipsoids are drawn at the 35% probability level. H atoms are represented by spheres of arbitrary size.