Table 3. *Short intramolecular distances* (Å)

C(1)···C(9)	3·196 (2)	C(9)···C(10)	3·091 (2)
C(2)···C(11)	3·244 (3)	C(9)···C(11)	3·361 (3)
C(3)···C(5)	2·979 (2)	C(10)···C(10′)	2·882 (2)
C(4)···C(10)	3·015 (2)	C(10)···C(15′)	3·213 (2)
C(4)···C(11)	3·300 (2)		

indicate absence of electronic interactions between the phenyl rings. In compound (2) these angles range from 62·0 to 70·7°. The largest deviations from the best planes in (1) are 0·008 and 0·009 Å in rings *A* and *B*, respectively, whereas in the central ring this value is 0·02 Å [torsion angles C(10)—C(1)—C(1′)—C(10′) 9·7 (2)° and C(4)—C(2)—C(1)—C(10) −3·7 (2)°].

The bond distances in the benzene rings range from 1·373 (3) to 1·415 (2) Å. Despite the repulsion of the phenyl rings (short intramolecular distances are given in Table 3), the bond distances are not lengthened significantly. The bond angles C(1)—C(2)—C(4) [124·3 (1)°] and C(3)—C(2)—C(4) [117·4 (1)°], however, are influenced by this repulsion effect.

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Structures of 5-Bromobicyclo[6.3.0]undecane-2,6-dione (I) and 2-Methyl-4,5,6,6a,8,9,9a,10-octahydro-7*H*-cyclopentacycloocteno[5,6-*b*]furan-6-one (II)

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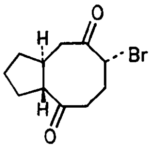
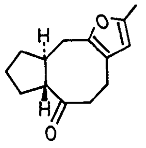
Abstract. $C_{11}H_{15}BrO_2$ (I): $M_r = 259{\cdot}1$, monoclinic, $P2_1/c$, $a = 6{\cdot}401$ (1), $b = 16{\cdot}493$ (3), $c = 10{\cdot}928$ (2) Å, $\beta = 111{\cdot}02$ (2)°, $V = 1076{\cdot}8$ (4) Å^3, $Z = 4$, $D_m = 1{\cdot}58$ (3), $D_x = 1{\cdot}60$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 3{\cdot}75$ mm^{-1}, $F(000) = 528$, $T = 295$ (1) K, $R = 0{\cdot}046$ for 1568 observed unique reflections. $C_{14}H_{18}O_2$ (II): $M_r = 218{\cdot}3$, monoclinic, $P2_1/c$, $a = 4{\cdot}939$ (1), $b = 12{\cdot}900$ (2), $c = 19{\cdot}024$ (2) Å, $\beta = 93{\cdot}17$ (2)°, $V = 1210{\cdot}2$ (4) Å^3, $Z = 4$, $D_m = 1{\cdot}20$ (3), $D_x = 1{\cdot}19$ Mg m^{-3}, Mo $K\alpha$, $\mu = 0{\cdot}07$ mm^{-1}, $F(000) = 472$, $T = 300$ (1) K, $R = 0{\cdot}041$ for 1188 observed unique reflections. The ring fusion of the five- and eight-membered rings in both (I) and (II) is *trans*. The boat–chair conformation of the eight-membered rings is somewhat distorted because of steric repulsion between C=O and C—Br bonds in (I) and the double bond in the eight-membered ring of (II).

Introduction. Compounds (I) and (II) were synthesized by one of the authors (MU) in the course of preparation of the linearly fused 5–8–5 ring system which is the partial skeleton of some sesquiterpenes and diterpenes. NMR spectroscopy is not very useful in the structural study of these compounds, because the eight-membered ring takes several conformations. The structures of (I) and (II) have been determined to elucidate the ring junction and to compare the conformation of the eight-membered ring with the related compounds 7-bromo-4,4-dimethylbicyclo[6.3.0]undecane-2,6-dione (III; Umehara, Takayanagi, Ogura & Hishida, 1978) and cyclooctane-1,5-dione (IV; Miller & McPhail, 1979).


(I)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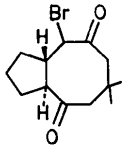
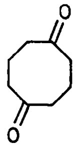

(III)


(IV)

Begley, Mellor & Pattenden (1983) determined the structure of another 5–8 fused-ring compound, bicyclo-[6.3.0]undecane-2,6-dione, to confirm that the ring junction is *trans*. It is noted that the four H atoms at the 2,6-positions of cyclooctane are replaced by carbonyl groups in these compounds to alleviate transannular H···H interactions.

Table 1. *Positional parameters* (× 10^4; *for* Br × 10^5) *and equivalent isotropic thermal parameters* (*Hamilton*, 1959)

	x	y	z	B_{eq}(Å^2 × 10)
(I)				
Br	34167 (8)	5988 (3)	8368 (5)	46
O(1)	−2309 (5)	2696 (2)	−1467 (3)	37
O(2)	3608 (5)	2372 (2)	291 (3)	37
C(1)	−1533 (6)	3182 (2)	682 (3)	22
C(2)	−1829 (6)	2529 (2)	−330 (4)	23
C(3)	−1588 (6)	1648 (2)	86 (4)	30
C(4)	−5 (7)	1426 (2)	1463 (4)	33
C(5)	2481 (7)	1488 (3)	1676 (4)	31
C(6)	3063 (6)	2300 (3)	1222 (4)	28
C(7)	2722 (7)	3026 (3)	1977 (4)	33
C(8)	835 (6)	3566 (2)	1146 (3)	25
C(9)	530 (8)	4341 (3)	1809 (4)	39
C(10)	−1896 (8)	4593 (3)	1159 (5)	44
C(11)	−3028 (7)	3925 (2)	191 (4)	32
(II)				
O(1)	425 (4)	−769 (2)	1974 (1)	59
O(2)	565 (4)	2700 (1)	1147 (1)	43
C(1)	3872 (5)	243 (2)	2557 (1)	37
C(2)	2636 (5)	−360 (2)	1939 (1)	39
C(3)	4191 (5)	−467 (2)	1282 (1)	44
C(4)	5378 (5)	541 (2)	1023 (1)	44
C(5)	3306 (5)	1382 (2)	918 (1)	39
C(6)	2543 (5)	2039 (2)	1419 (1)	39
C(7)	3375 (6)	2179 (2)	2179 (1)	43
C(8)	2485 (5)	1311 (2)	2657 (1)	40
C(9)	3140 (7)	1535 (2)	3436 (1)	58
C(10)	2823 (9)	495 (3)	3788 (2)	79
C(11)	3602 (7)	−308 (2)	3269 (2)	56
C(12)	1707 (5)	1638 (2)	292 (1)	43
C(13)	113 (5)	2436 (2)	449 (1)	43
C(14)	−1941 (6)	3066 (2)	49 (2)	57

Experimental. (I): Colorless plate-like crystals from an acetone solution. D_m measured by flotation in aqueous solution of potassium iodide. Crystal size 0·3 × 0·5 × 0·5 mm; Rigaku AFC-5 four-circle diffractometer; Mo $K\alpha$ radiation monochromatized by graphite plate. Laue group $2/m$. Unit-cell parameters from 20 reflections ($20 < 2\theta < 26°$). Intensities measured up to $2\theta = 55°$, $-8 \leq h \leq 8$, $0 \leq k \leq 21$, $0 \leq l \leq 14$, θ–2θ scan, scan speed 6° min^{-1} in θ. 2603 reflections collected, 1654 observed [$|F_o| > 3\sigma(|F_o|)$], 736 zero, 213 unobserved, 1568 unique ($R_{\rm int} = 0·027$). Mean ratio of $|F_o|$ of five standard reflections, $0·98 < \sum(|F_o|/|F_o|_{\rm initial})/5 < 1·01$. Absorption correction ($0·175 < A < 0·330$). Structure solved by heavy-atom method. Non-H atoms refined with anisotropic thermal parameters, all H atoms located in difference synthesis, and refined isotropically. Function $\sum w(|F_o| - |F_c|)^2$ minimized. Final $R = 0·046$, $wR = 0·053$, $w^{-1} = \sigma^2(F) + (0·015|F_o|)^2$, $S = 1·51$, $(\Delta/\sigma)_{\rm max} = 0·5$, $-0·7 < \Delta\rho < 0·4$ e Å^{-3}. (II): Colorless prismatic crystals from n-hexane solution; crystal size 0·3 × 0·5 × 0·6 mm. Laue group $2/m$. Unit-cell parameters from 20 reflections ($20 < 2\theta < 29°$). Intensities measured up to $2\theta = 50°$, $0 \leq h \leq 5$, $0 \leq k \leq 15$, $-22 \leq l \leq 22$. 2394 reflections collected, 1353 observed, 1017 zero, 24 unobserved, 1188 unique ($R_{\rm int} = 0·010$). Mean ratio of $|F_o|$ of five standard reflections $0·93 < \sum(|F_o|/|F_o|_{\rm initial})/5 < 1·01$. No absorption correction. Structure solved by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). All H atoms located in difference synthesis and refined. Final $R = 0·041$, $wR = 0·053$, $S = 2·27$, $(\Delta/\sigma)_{\rm max} = 0·4$, $-0·2 < \Delta\rho < 0·2$ e Å^{-3}.* Complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); *Universal Crystallographic Computation Program System UNICS*III (Sakurai & Kobayashi, 1979).

Discussion. Table 1 lists atomic coordinates and Table 2 bond lengths and bond angles. Fig. 1 shows perspective views of the molecules. The ring fusion in both (I) and (II) is *trans*. The torsion angle H(C1)—C(1)—C(8)—H(C8) in (I) and (II) is 165·6 (2·8) and 162·9 (1·7)°, respectively. The eight-membered rings take a boat–chair form, which is the most stable conformation predicted by molecular-mechanics calculations (Hendrickson, 1967; Engler, Andose & Schleyer, 1973). The C(1)—C(8) bond of (I) and (II) is longer by *ca* 0·030 (5) Å than the other C—C bonds, suggesting a strain in these 5–8 fused-ring structures. The only intermolecular distance less than the sum of

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* Lists of structure factors, anisotropic thermal parameters, atomic parameters for H atoms, bond lengths and bond angles involving H atoms and torsion angles for (I) and (II) have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43853 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å), *bond angles* (°) *and transannular* H···H *distances* (Å)

(I)			
Br—C(5)	1·935 (5)	C(2)—C(1)—C(8)	112·5 (3)
O(1)—C(2)	1·200 (5)	C(2)—C(1)—C(11)	114·7 (3)
O(2)—C(6)	1·194 (6)	C(8)—C(1)—C(11)	101·7 (3)
C(1)—C(2)	1·506 (5)	O(1)—C(2)—C(1)	120·9 (3)
C(1)—C(8)	1·551 (5)	O(1)—C(2)—C(3)	119·5 (3)
C(1)—C(11)	1·529 (5)	C(1)—C(2)—C(3)	119·6 (3)
C(2)—C(3)	1·514 (5)	C(2)—C(3)—C(4)	119·1 (3)
C(3)—C(4)	1·527 (5)	C(3)—C(4)—C(5)	114·9 (4)
C(4)—C(5)	1·527 (6)	Br—C(5)—C(4)	111·1 (3)
C(5)—C(6)	1·520 (7)	Br—C(5)—C(6)	111·1 (3)
C(6)—C(7)	1·514 (7)	C(4)—C(5)—C(6)	111·9 (4)
C(7)—C(8)	1·513 (5)	O(2)—C(6)—C(5)	123·1 (5)
C(8)—C(9)	1·516 (6)	O(2)—C(6)—C(7)	121·9 (5)
C(9)—C(10)	1·516 (7)	C(5)—C(6)—C(7)	114·8 (4)
C(10)—C(11)	1·519 (6)	C(6)—C(7)—C(8)	112·2 (3)
H(C1)···H(C4)2	2·20 (4)	C(1)—C(8)—C(7)	115·6 (3)
H(C1)···H(C7)2	2·58 (4)	C(1)—C(8)—C(9)	102·8 (3)
H(C4)2···H(C7)2	2·47 (5)	C(7)—C(8)—C(9)	114·9 (3)
		C(8)—C(9)—C(10)	107·0 (3)
		C(9)—C(10)—C(11)	106·3 (4)
		C(1)—C(11)—C(10)	104·8 (3)
(II)			
O(1)—C(2)	1·218 (3)	C(6)—O(2)—C(13)	106·5 (2)
O(2)—C(6)	1·376 (3)	C(2)—C(1)—C(8)	112·9 (2)
O(2)—C(13)	1·376 (3)	C(2)—C(1)—C(11)	113·3 (2)
C(1)—C(2)	1·510 (4)	C(8)—C(1)—C(11)	104·0 (2)
C(1)—C(8)	1·555 (4)	O(1)—C(2)—C(1)	120·4 (2)
C(1)—C(11)	1·542 (4)	O(1)—C(2)—C(3)	120·2 (2)
C(2)—C(3)	1·510 (4)	C(1)—C(2)—C(3)	119·3 (2)
C(3)—C(4)	1·520 (4)	C(2)—C(3)—C(4)	114·4 (2)
C(4)—C(5)	1·497 (4)	C(3)—C(4)—C(5)	113·0 (2)
C(5)—C(6)	1·345 (4)	C(4)—C(5)—C(6)	125·3 (2)
C(5)—C(12)	1·430 (4)	C(4)—C(5)—C(12)	128·3 (2)
C(6)—C(7)	1·493 (4)	C(6)—C(5)—C(12)	106·3 (2)
C(7)—C(8)	1·521 (4)	O(2)—C(6)—C(5)	110·1 (2)
C(8)—C(9)	1·528 (4)	O(2)—C(6)—C(7)	116·2 (2)
C(9)—C(10)	1·511 (5)	C(5)—C(6)—C(7)	133·6 (2)
C(10)—C(11)	1·496 (5)	C(6)—C(7)—C(8)	114·6 (2)
C(12)—C(13)	1·339 (4)	C(1)—C(8)—C(7)	115·8 (2)
C(13)—C(14)	1·477 (4)	C(1)—C(8)—C(9)	102·4 (2)
H(C1)···H(C4)2	2·29 (3)	C(7)—C(8)—C(9)	112·8 (2)
H(C1)···H(C7)2	2·47 (3)	C(8)—C(9)—C(10)	103·9 (2)
H(C4)2···H(C7)2	2·63 (3)	C(9)—C(10)—C(11)	106·6 (3)
		C(1)—C(11)—C(10)	107·4 (2)
		C(5)—C(12)—C(13)	107·3 (2)
		O(2)—C(13)—C(12)	109·8 (2)
		O(2)—C(13)—C(14)	115·5 (2)
		C(12)—C(13)—C(14)	134·7 (3)

the van der Waals radius is O(1)···C(6) (x, $\frac{1}{2}-y$, $\frac{1}{2}+z$) of 3·128 (4) Å in (I). Fig. 2 shows the side views of the eight-membered rings in (I)–(IV). (IV) shows a typical boat–chair form, having a pseudo-mirror symmetry. (III) is similar to (IV), but (I) and (II) are somewhat distorted. The deviation parameter, ΔBC, which indicates the magnitude of the deviation of the torsion angles from the symmetrical boat–chair form (Miller & McPhail, 1979), is 18·7, 26·1, 4·8 and 2·6°, for (I), (II), (III) and (IV), respectively (Table 3). The torsion angle C(4)—C(5)—C(6)—C(7) is 64·2 (5) and 0·7 (5)° for (I) and (II), respectively. The corresponding value on the other side of the ring, C(1)—C(2)—C(3)—C(4), is −30·3 (5) and −47·4 (3)°, respectively. The origin of the deformation of the eight-membered ring in (I) is a steric repulsion between O(2) and Br. The torsion angle Br—C(5)—C(6)—O(2) is 13·7 (5)°, whereas the Br—C—C—O torsion angle in (III) is 110·8° (Umehara *et al.*, 1978). The deformation in (II) is largely due to a double bond between C(5) and C(6). The cyclopentane

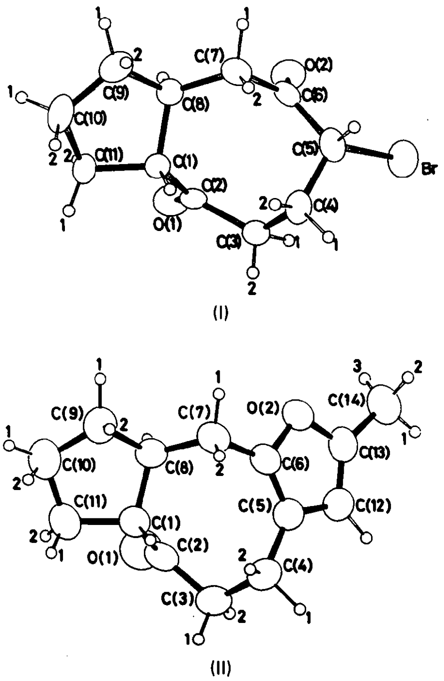


Fig. 1. *ORTEP* drawings (Johnson, 1965) of (I) and (II) with 50% probability ellipsoids. H atoms are represented by circles of radius 0·08 Å.

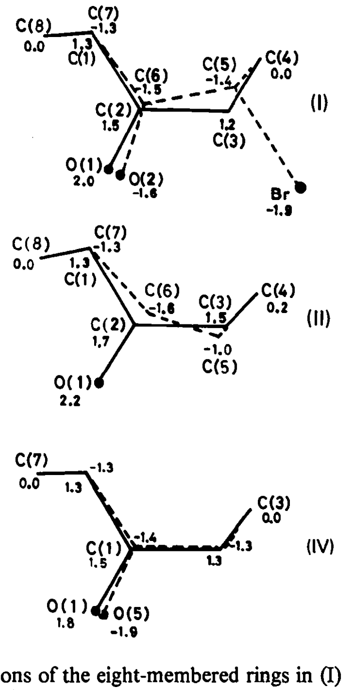
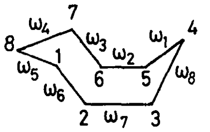


Fig. 2. Projections of the eight-membered rings in (I) and (II) along the C(1)···C(7) vector. The corresponding projection of (IV) is also shown. The numerical values are the relative heights of the atoms. Solid lines show the front part of the ring, and dashed lines the back.

Table 3. *Torsion angles (°) for eight-membered rings*



	ω_4	ω_3	ω_2	ω_1	
	ω_5	ω_6	ω_7	ω_8	ΔBC (°)*
(I)	65·2	−112·3	64·2	50·4	18·7
	−70·2	98·8	−30·3	−72·7	
(II)	68·0	−69·9	0·7	86·6	26·1
	−80·5	−107·3	−47·4	−54·0	
(IV)	69·2	107·5	47·5	62·8	2·6
	−67·3	105·0	−50·1	−59·6	

* $\Delta BC = (|\omega_1 + \omega_8| + |\omega_2 + \omega_7| + |\omega_3 + \omega_6| + |\omega_4 + \omega_5|)/4$.

rings take envelope conformations, out-of-plane atoms being C(1) in (I) and C(8) in (II). The furan ring in (II) is essentially planar with deviations less than 0·005 Å for each C atom from the least-squares plane.

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Structure of 3,4-Diphenyl-1,2,4-oxadiazol-5-one

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Abstract. $C_{14}H_{10}N_2O_2$, $M_r = 238{\cdot}24$, monoclinic, $P2_1/a$, $a = 13{\cdot}064\,(3)$, $b = 5{\cdot}903\,(2)$, $c = 15{\cdot}341\,(3)$ Å, $\beta = 102{\cdot}22\,(2)°$, $V = 1156{\cdot}2\,(8)$ Å^3, $Z = 4$, $D_x = 1{\cdot}37$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}0876$ mm^{-1}, $F(000) = 496$, $T = 296$ K, final $R(F) = 0{\cdot}032$ for 1020 observed unique reflections. The structure contains a planar oxadiazole heterocycle showing aromatic character and two phenyl rings twisted 35 and 66° from the heterocycle plane.

Introduction. The 1,2,4-oxadiazol-5-one derivatives have many biological and pharmaceutical properties (Breuer, 1976). The title compound was synthesized from the action of isocyanates on hydroximates (Bel Hadj Amor & Baccar, 1986). It was characterized by IR and ^{1}H NMR spectroscopy and elemental analysis. Many conformations are possible for the heterocycle, so we have undertaken a study by X-ray diffraction to determine the crystal structure and the exact geometry of the molecule.

Experimental. Prismatic transparent crystals were obtained by slow evaporation from a solution of the material in methanol. A sample *ca* 0·60 × 0·40 × 0·20 mm was chosen for X-ray diffraction. Data were collected on a CAD-4 diffractometer, with graphite-monochromated Mo $K\alpha$ radiation and $\omega/2\theta$ scan. Cell parameters from setting angles of 23 reflections with $9{\cdot}5 < \theta < 12°$. Hemisphere up to $2\theta = 46°$, $[(\sin\theta)/\lambda]_{\max} = 0{\cdot}55$ Å^{-1}, range of hkl: h: 0→14, k: ±6, l: ±16. Two standard reflections measured every 100 reflections remained stable. Total number of reflections 3260. Averaging of equivalent reflections ($R_{\text{int}} = 0{\cdot}039$) resulted in 1615 independent reflections for structure determination. Only 1020 with $I > 3\sigma(I)$ were used for structure refinement. The intensities were corrected for Lorentz–polarization effects but for neither absorption nor extinction. Structure determination by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) and anisotropic refinement on F by *SHELX*76 (Sheldrick, 1976). All H atoms were found

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