Table 5. *Some relevant torsion angles* (°)

Cyclohexanone ring		Cyclohexanedione ring		Exocyclic	
C(8)–C(2)–C(1)–C(21)	−64·8 (3)	C(8)–C(13)–C(12)–C(11)	−18·8 (7)	C(8)–C(14)–C(15)–O(3)	56·9 (3)
C(2)–C(1)–C(21)–C(20)	33·1 (4)	C(13)–C(12)–C(11)–C(10)	9·2 (11)	C(8)–C(14)–C(15)–C(19)	−123·9 (4)
C(1)–C(21)–C(20)–C(14)	28·0 (4)	C(12)–C(11)–C(10)–C(9)	11·8 (11)	C(20)–C(14)–C(15)–C(19)	3·6 (5)
C(21)–C(20)–C(14)–C(8)	−63·0 (3)	C(11)–C(10)–C(9)–C(8)	−22·7 (7)	C(8)–C(2)–C(3)–O(2)	72·8 (3)
C(20)–C(14)–C(8)–C(2)	31·3 (3)	C(10)–C(9)–C(8)–C(13)	12·5 (5)	C(8)–C(2)–C(3)–C(7)	−104·6 (4)
C(14)–C(8)–C(2)–C(1)	30·5 (3)	C(9)–C(8)–C(13)–C(12)	7·6 (4)	C(1)–C(2)–C(3)–C(7)	22·9 (5)
				C(1)–C(2)–C(3)–O(2)	−159·7 (3)

C(11)–C(10)–C(9) [118·5 (4)°] are larger than expected for tetrahedral angles. These observations may be related to the large thermal parameters of C(10), C(11) and C(12), possibly indicating some disorder in the cyclohexanedione ring, the nature of which has not been analysed further.

Some endocyclic and exocyclic torsion angles are listed in Table 5. The torsion angles in the cyclohexanone ring (−64·8, 33·1, 28·0, −63·0, 31·3, 30·5°) are near to the ideal twist-boat torsion angles −70·6, 33·2, 33·2, −70·6, 33·2, 33·2° (Pazdernik, Brisse & Rivest, 1977); the cyclohexanedione ring also has the twist-boat configuration, but less pronounced.

The methylfuryl rings are flat; the maximum deviation of the atoms from the least-squares plane is 0·004 Å in both rings. Intermolecular distances correspond to van der Waals contacts and no strong intermolecular interactions are apparent.

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cis-Bicyclo[3.2.0]hept-2-en-6-one *p*-Nitrophenylhydrazone

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Abstract. $C_{13}H_{13}N_3O_2$, M_r = 243·26, monoclinic, $C2/c$, a = 33·55 (5), b = 4·72 (5), c = 31·11 (5) Å, β = 97·83 (2)° from diffractometer measurements (Mo $K\bar{\alpha}$ radiation), V = 4876 Å^3, Z = 16, D_c = 1·32 Mg m^{-3}, $F(000)$ = 2048, μ = 0·055 mm^{-1}, approximate crystal dimensions 0·4 × 0·2 × 0·1 mm. The molecules are linked into hydrogen-bonded tetramers.

Introduction. Systematic absences (from precession photographs) hkl: $h + k$ odd and $h0l$: l odd indicated space group $C2/c$. Data were collected for $h0$–$3l$ with θ_{max} = 23·75° on a Stoe STADI-2 two-circle diffractometer (graphite-monochromated Mo $K\bar{\alpha}$ radiation). This gave 2968 data of which 1301 unique reflexions with $I > 3\sigma(I)$ were used in subsequent calculations. Lorentz and polarization corrections (but none for extinction or absorption) were applied, and the data scaled by a Wilson plot. The structure was solved by direct phasing methods with *SHELX* 76 (Sheldrick, 1976), which was used for all calculations. Complex neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Weighted full-matrix least-squares refinement (including isotropic H atoms) converged at R = 0·066 for

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *with e.s.d.'s in parentheses*

	x	*y*	*z*
C(1*A*)	2073 (2)	−2712 (19)	1733 (2)
C(1*B*)	−1102 (2)	14507 (18)	2078 (2)
C(2*A*)	2297 (3)	−653 (24)	2045 (2)
C(2*B*)	−1254 (2)	12800 (23)	2434 (3)
C(3*A*)	2645 (2)	142 (22)	1925 (2)
C(3*B*)	−964 (3)	11764 (23)	2714 (2)
C(4*A*)	2734 (2)	−1192 (18)	1521 (2)
C(4*B*)	−554 (2)	12545 (21)	2617 (2)
C(5*A*)	2332 (2)	−2691 (17)	1352 (2)
C(5*B*)	−638 (2)	14072 (19)	2179 (2)
C(6*A*)	2014 (1)	−818 (16)	1109 (1)
C(6*B*)	−663 (2)	12239 (17)	1787 (2)
C(7*A*)	1720 (2)	−1330 (17)	1431 (2)
C(7*B*)	−1103 (2)	12862 (17)	1641 (2)
C(10*A*)	1684 (1)	4276 (15)	328 (1)
C(10*B*)	−245 (1)	7347 (15)	1134 (1)
C(11*A*)	2017 (1)	4662 (15)	105 (1)
C(11*B*)	149 (1)	7042 (16)	1352 (1)
C(12*A*)	2001 (1)	6580 (15)	−231 (1)
C(12*B*)	405 (1)	5165 (17)	1192 (2)
C(13*A*)	1649 (2)	8153 (16)	−335 (2)
C(13*B*)	281 (2)	3623 (17)	821 (2)
C(14*A*)	1315 (1)	7808 (16)	−117 (2)
C(14*B*)	−104 (2)	3968 (18)	594 (2)
C(15*A*)	1334 (1)	5891 (16)	210 (2)
C(15*B*)	−365 (1)	5856 (16)	752 (1)
N(8*A*)	2035 (1)	813 (12)	791 (1)
N(8*B*)	−399 (1)	10653 (13)	1661 (1)
N(9*A*)	1694 (1)	2437 (12)	667 (1)
N(9*B*)	−515 (1)	9127 (12)	1287 (1)
N(16*A*)	1630 (1)	10199 (15)	−682 (2)
N(16*B*)	557 (2)	1617 (16)	664 (2)
O(17*A*)	1931 (1)	10620 (12)	−865 (1)
O(17*B*)	886 (1)	1208 (14)	898 (1)
O(18*A*)	1319 (1)	11618 (11)	−787 (1)
O(18*B*)	461 (1)	473 (15)	315 (2)
H(1*A*)*A*	2007	−4378	1941
H(1*B*)*A*	−1266	16456	2069
H(11*A*)	2289	3464	196
H(11*B*)	251	8271	1641
H(12*A*)	2252	6836	−411
H(12*B*)	705	4809	1352
H(14*A*)	1054	9075	−208
H(14*B*)	−190	2816	299
H(15*A*)	1072	5496	370
H(15*B*)	−664	6058	592
H(2*A*)*A*	2209	76	2341
H(2*B*)*A*	−1574	12416	2437
H(3*A*)*A*	2845	1780	2093
H(3*B*)*A*	−1024	10540	2985
H(4*A*)*A*	2807	332	1284
H(4*A*)*B*	2979	−2700	1585
H(4*B*)*A*	−407	13909	2864
H(4*B*)*B*	−373	10651	2593
H(5*A*)*A*	2423	−4564	1193
H(5*B*)*A*	−420	15737	2203
H(7*A*)*A*	1590	564	1550
H(7*A*)*B*	1484	−2806	1316
H(7*B*)*A*	−1164	14223	1352
H(7*B*)*B*	−1297	11045	1595
H(9*A*)*A*	1436	2221	832
H(9*B*)*A*	−817	9321	1117

1301 observed reflexions ($R = \sum ||F_o| - |F_c|| / \sum |F_o|$); $R_w = 0{\cdot}047$ $\{R_w = (\sum ||F_o| - |F_c||\, w^{1/2}) / (\sum |F_o|\, w^{1/2})$, $w = 3{\cdot}75/[\sigma^2(F_o) + 0{\cdot}00003F_o^2]\}$. In the final cycle all shifts in parameters were less than their e.s.d.'s. Positional parameters are given in Table 1, bond distances and angles in Table 2.*

Discussion. Bicyclo[3.2.0]hept-2-en-6-one (II) is an important intermediate in a recent total synthesis of prostaglandins (Dimsdale, Newton, Rainey, Webb, Lee & Roberts, 1977). It shows a remarkable regio- and stereoselectivity in adding the elements of HOBr which is not found in the lactone (III). We are undertaking a survey of the geometries of derivatives of (II) and (III) to try to isolate steric factors which may affect the

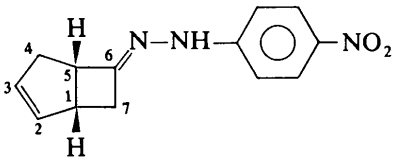
* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34414 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

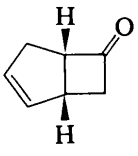
C(1*A*)–C(2*A*)	1·502 (12)	C(10*A*)–N(9*A*)	1·361 (7)
C(1*A*)–C(5*A*)	1·562 (10)	C(10*B*)–C(11*B*)	1·407 (6)
C(1*A*)–C(7*A*)	1·552 (9)	C(10*B*)–C(15*B*)	1·392 (7)
C(1*B*)–C(2*B*)	1·513 (12)	C(10*B*)–N(9*B*)	1·368 (7)
C(1*B*)–C(5*B*)	1·559 (9)	C(11*A*)–C(12*A*)	1·378 (8)
C(1*B*)–C(7*B*)	1·567 (10)	C(11*B*)–C(12*B*)	1·373 (9)
C(2*A*)–C(3*A*)	1·327 (13)	C(12*A*)–C(13*A*)	1·394 (8)
C(2*B*)–C(3*B*)	1·307 (11)	C(12*B*)–C(13*B*)	1·380 (9)
C(3*A*)–C(4*A*)	1·473 (10)	C(13*A*)–C(14*A*)	1·395 (8)
C(3*B*)–C(4*B*)	1·493 (11)	C(13*A*)–N(16*A*)	1·443 (9)
C(4*A*)–C(5*A*)	1·547 (9)	C(13*B*)–C(14*B*)	1·393 (7)
C(4*B*)–C(5*B*)	1·534 (10)	C(13*B*)–N(16*B*)	1·455 (10)
C(5*A*)–C(6*A*)	1·508 (9)	C(14*A*)–C(15*A*)	1·357 (9)
C(5*B*)–C(6*B*)	1·488 (10)	C(14*B*)–C(15*B*)	1·385 (9)
C(6*A*)–C(7*A*)	1·517 (8)	N(8*A*)–N(9*A*)	1·388 (6)
C(6*A*)–N(8*A*)	1·262 (7)	N(8*B*)–N(9*B*)	1·378 (6)
C(6*B*)–C(7*B*)	1·513 (8)	N(16*A*)–O(17*A*)	1·241 (7)
C(6*B*)–N(8*B*)	1·261 (8)	N(16*A*)–O(18*A*)	1·247 (7)
C(10*A*)–C(11*A*)	1·407 (6)	N(16*B*)–O(17*B*)	1·249 (7)
C(10*A*)–C(15*A*)	1·406 (7)	N(16*B*)–O(18*B*)	1·218 (8)

C(5*A*)–C(1*A*)–C(2*A*)	101·8 (6)	N(9*A*)–C(10*A*)–C(15*A*)	118·5 (4)
C(7*A*)–C(1*A*)–C(2*A*)	113·2 (7)	C(15*B*)–C(10*B*)–C(11*B*)	120·2 (5)
C(7*A*)–C(1*A*)–C(5*A*)	89·8 (5)	N(9*B*)–C(10*B*)–C(11*B*)	121·3 (5)
C(5*B*)–C(1*B*)–C(2*B*)	102·1 (6)	N(9*B*)–C(10*B*)–C(15*B*)	118·5 (4)
C(7*B*)–C(1*B*)–C(2*B*)	114·3 (7)	C(12*A*)–C(11*A*)–C(10*A*)	120·5 (5)
C(7*B*)–C(1*B*)–C(5*B*)	89·6 (5)	C(12*B*)–C(11*B*)–C(10*B*)	118·9 (5)
C(3*A*)–C(2*A*)–C(1*A*)	112·5 (7)	C(13*A*)–C(12*A*)–C(11*A*)	118·1 (5)
C(3*B*)–C(2*B*)–C(1*B*)	113·1 (7)	C(13*B*)–C(12*B*)–C(11*B*)	120·8 (5)
C(4*A*)–C(3*A*)–C(2*A*)	114·0 (7)	C(14*A*)–C(13*A*)–C(12*A*)	122·4 (6)
C(4*B*)–C(3*B*)–C(2*B*)	113·3 (7)	N(16*A*)–C(13*A*)–C(12*A*)	118·6 (5)
C(5*A*)–C(4*A*)–C(3*A*)	102·6 (5)	N(16*A*)–C(13*A*)–C(14*A*)	119·0 (5)
C(5*B*)–C(4*B*)–C(3*B*)	103·5 (5)	C(14*B*)–C(13*B*)–C(12*B*)	121·0 (6)
C(4*A*)–C(5*A*)–C(1*A*)	107·2 (5)	N(16*B*)–C(13*B*)–C(12*B*)	119·3 (5)
C(6*A*)–C(5*A*)–C(1*A*)	87·4 (5)	N(16*B*)–C(13*B*)–C(14*B*)	119·7 (6)
C(6*A*)–C(5*A*)–C(4*A*)	115·2 (6)	C(15*A*)–C(14*A*)–C(13*A*)	119·0 (5)
C(4*B*)–C(5*B*)–C(1*B*)	107·3 (6)	C(15*B*)–C(14*B*)–C(13*B*)	118·8 (5)
C(6*B*)–C(5*B*)–C(1*B*)	88·2 (5)	C(14*A*)–C(15*A*)–C(10*A*)	120·5 (5)
C(6*B*)–C(5*B*)–C(4*B*)	116·0 (7)	C(14*B*)–C(15*B*)–C(10*B*)	120·3 (4)
C(7*A*)–C(6*A*)–C(5*A*)	93·1 (5)	N(9*A*)–N(8*A*)–C(6*A*)	115·1 (4)
N(8*A*)–C(6*A*)–C(5*A*)	129·8 (5)	N(9*B*)–N(8*B*)–C(6*B*)	116·3 (4)
N(8*A*)–C(6*A*)–C(7*A*)	136·3 (6)	N(8*A*)–N(9*A*)–C(10*A*)	119·9 (4)
C(7*B*)–C(6*B*)–C(5*B*)	94·4 (5)	N(8*B*)–N(9*B*)–C(10*B*)	119·3 (4)
N(8*B*)–C(6*B*)–C(5*B*)	129·5 (5)	O(17*A*)–N(16*A*)–C(13*A*)	119·7 (5)
N(8*B*)–C(6*B*)–C(7*B*)	136·0 (6)	O(18*A*)–N(16*A*)–C(13*A*)	120·3 (5)
C(6*A*)–C(7*A*)–C(1*A*)	87·5 (4)	O(18*A*)–N(16*A*)–O(17*A*)	120·0 (6)
C(6*B*)–C(7*B*)–C(1*B*)	87·1 (4)	O(17*B*)–N(16*B*)–C(13*B*)	117·3 (6)
C(15*A*)–C(10*A*)–C(11*A*)	119·6 (5)	O(18*B*)–N(16*B*)–C(13*B*)	118·9 (5)
N(9*A*)–C(10*A*)–C(11*A*)	121·9 (5)	O(18*B*)–N(16*B*)–O(17*B*)	123·8 (7)

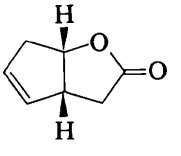
reaction. Since (II) is a liquid at room temperature we have studied the crystalline derivative (I) to investigate the conformations of the fused rings.



(I)



(II)



(III)

There are two independent molecules in the asymmetric unit (Fig. 1), hydrogen-bonded into a centrosymmetric tetramer [N(9*A*)–H···O(17*B*) = 2·96 Å; N(9*B*)–H···O(18*A*)(x, $2-y$, z) = 2·94 Å]. The bond lengths and angles agree well between the two molecules, but the conformations (Table 3) of the fused systems, although similar, are significantly different.

Table 3. *Torsion angles* (°) (*e.s.d.'s ca* 0·8°) *for selected bonds in molecules A and B*

Note that *A* has the chirality shown in (I) whereas *B* is the enantiomer.

	A	*B*
C(1)–C(2)–C(3)–C(4)	−1	1
C(2)–C(3)–C(4)–C(5)	9	−6
C(3)–C(4)–C(5)–C(1)	−13	9
C(4)–C(5)–C(1)–C(2)	13	−8
C(5)–C(1)–C(2)–C(3)	−8	5
C(1)–C(5)–C(6)–C(7)	−11	6
C(5)–C(6)–C(7)–C(1)	11	−6
C(6)–C(7)–C(1)–C(5)	−11	6
C(7)–C(1)–C(5)–C(6)	11	−6

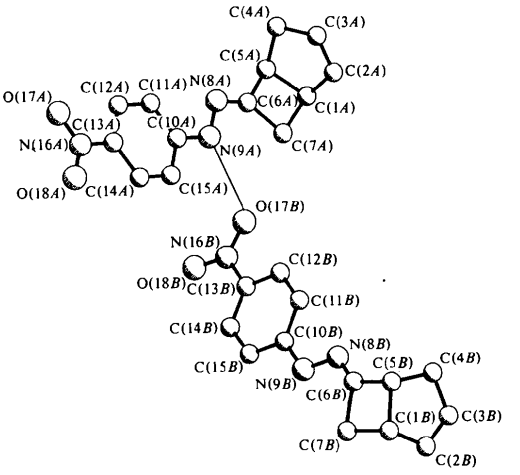


Fig. 1. The two independent molecules of (I) linked by a hydrogen bond. (Hydrogen atoms are omitted from the diagram.)

Each five-membered ring is a shallow envelope, with C(5) below the plane of C(1), C(2), C(3), C(4) when the molecule is oriented as in (II), and the cyclobutane rings are puckered. The degree of puckering is about 50% higher in molecule *A* than in *B*, and this suggests that the molecule is somewhat flexible, since packing forces are unlikely to be very different for the two cases.

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