Table 4. *Least-squares planes*

Plane	Atoms defining the plane	σ_s (Å)
1	C(1), C(6), C(7), C(8), C(9), C(10), C(11), O(3)	0·01064
2	C(1), C(2), O(3)	–
3	C(1), C(5), O(1)	–
4	C(1), C(2), C(4), C(5)	0·09742
5	C(2), C(3), C(4)	–

Distances of atoms from the planes (Å) (e.s.d.'s in parentheses)

Plane 1		Plane 2		Plane 4	
Br	0·036 (1)	C(3)	1·203 (9)	C(3)	–0·562 (9)
C(2)	–0·220 (8)	C(4)	2·467 (9)	O(1)	–0·583 (6)
C(3)	0·728 (9)	C(5)	2·386 (9)	O(2)	0·138 (8)
C(4)	2·160 (9)	C(6)	–0·371 (8)	O(3)	1·544 (6)
C(5)	2·368 (9)	C(7)	–0·681 (8)	C(6)	1·335 (8)
O(1)	1·248 (5)	C(8)	–0·931 (9)	C(7)	1·837 (8)
O(2)	3·073 (8)	Br	–1·341 (1)	Br	3·948 (1)

system (Foresti Serantoni, Merlini, Mongiorgi & Riva di Sanseverino, 1974; Fontaine, Dideberg & Dupont, 1975; Rodgers, Goaman & Blow, 1976).

The C(sp^3)—O(1) bond lengths in the pyran ring are approximately symmetric: 1·43 and 1·44 (1) Å. The bond angle at O(1) is 113·7 (7)°.

All the atoms of the benzofuran system except C(2) approximately form a plane (Table 4, plane 1). C(2) deviates from this plane by *ca* –0·2 Å. Plane 1 forms a dihedral angle of 166° with the plane of C(1), C(2) and O(3) (Table 4, plane 2), thus indicating the envelope shape of the dihydrofuran ring.

The tetrahydropyran ring reveals the boat conformation of $B_{O,3}$ type [for the nomenclature see Schwartz (1973)], deformed by the presence of C(4) with sp^2 hybridization. Three least-squares planes may be distinguished in the ring (Table 4, planes 3, 4 and 5). They form the following dihedral angles: planes 3–4 132° and planes 4–5 139°. The central plane 4 is somewhat distorted (the mean deviation of atoms is 0·04 Å), and forms a dihedral angle of 93° with plane 2, causing the nearly rectangular shape of the molecule.

The authors are much indebted to Professor Dr A. Zamojski and Dr G. Grynkiewicz for valuable discussions.

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Structure of Spiro[3,4-dihydro-2*H*-1,5-dioxadinaphtho[2,1-*f* : 1,2-*h*]cyclononene-3,1'-cyclopropane]

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Abstract. $C_{25}H_{20}O_2$, $M_r = 352·4$, orthorhombic, *Pbca*, $a = 10·690$ (1), $b = 10·792$ (1), $c = 31·131$ (4) Å, $U = 3591$ Å^3, $D_m = 1·303$ (2), $D_x = 1·303$ Mg m^{-3}, $Z = 8$, μ(Cu $K\alpha$) = 0·558 mm^{-1}. The structure was solved by direct methods and refined to $R = 0·068$ for 2612 counter-collected reflections. In the crystalline state the molecule adopts an asymmetrical conformation. The two naphthyl planes are twisted through an angle of 71·5°; this value differs significantly from that of the torsion angle at the single bond of the binaphthyl system inside the nine-membered ring (64·2°), showing a strain in the binaphthyl system.

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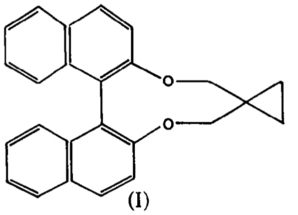
Introduction. The title compound (I) was synthesized and described by Smoliński & Jamrozik (1975). The crystals supplied were colourless and plate-like in shape. Unit-cell dimensions were obtained from a least-squares fit to the 15 strong reflections used in the auto-indexing procedure on a CAD-4 (Enraf–Nonius) diffractometer. The intensity measurements were carried out with graphite-monochromated Cu $K\alpha$ radiation up to $\theta = 65°$ using the ω–2θ scan mode and $(0{\cdot}6 + 0{\cdot}3 \tan\theta)°$ scan range. 2612 unique reflections with intensities greater than $\sigma(I)$ were obtained from a crystal of dimensions $0{\cdot}30 \times 0{\cdot}25 \times 0{\cdot}13$ mm. Lorentz and polarization corrections were applied. No correction was made for absorption. The structure was solved by direct methods and refined by full-matrix least squares to the final discrepancy indices $R = 0{\cdot}068$, $R_w = 0{\cdot}055$ and $R_G = 0{\cdot}065$, where $R = \sum |\Delta F|/\sum |F_o|$, $R_w = \sum w^{1/2}|\Delta F|/\sum w^{1/2}|F_o|$ and $R_G = [\sum w(\Delta F)^2/\sum w(F_o)^2]^{1/2}$. The calculations were performed with the *SHELX* 76 program (Sheldrick, 1976). The weighting scheme in the refinement was $w = [\sigma^2(F_o)]^{-1}$. Positions of all H atoms were found from a Fourier difference map. In the last cycle the ratios of the shifts to the e.s.d.'s were less than 0·1 for all refined parameters. The final atomic coordinates are given in Table 1.*

Table 1. *Fractional atomic coordinates* ($\times 10^4$; *for hydrogen atoms* $\times 10^3$) *with estimated standard deviations in parentheses*

	x	*y*	*z*
C(1)	3604 (3)	1319 (3)	1520 (1)
C(2)	2718 (4)	1238 (4)	1856 (1)
C(3)	2831 (4)	333 (4)	2157 (1)
C(4)	3812 (3)	−537 (3)	2140 (1)
C(5)	3941 (4)	−1486 (4)	2452 (1)
C(6)	4889 (5)	−2316 (4)	2431 (1)
C(7)	5761 (4)	−2269 (4)	2092 (1)
C(8)	5668 (4)	−1363 (3)	1785 (1)
C(9)	4691 (3)	−472 (3)	1796 (1)
C(10)	4574 (3)	496 (3)	1482 (1)
C(11)	5508 (3)	661 (3)	1129 (1)
C(12)	5598 (3)	−183 (3)	776 (1)
C(13)	4784 (4)	−1220 (3)	732 (1)
C(14)	4818 (4)	−1945 (4)	370 (1)
C(15)	5684 (4)	−1694 (4)	41 (1)
C(16)	6505 (4)	−748 (4)	82 (1)
C(17)	6480 (3)	38 (3)	446 (1)
C(18)	7267 (4)	1089 (4)	475 (1)
C(19)	7153 (3)	1898 (3)	803 (1)
C(20)	6254 (3)	1702 (3)	1127 (1)
O(1)	3465 (2)	2221 (2)	1210 (1)
O(2)	6150 (2)	2518 (2)	1470 (1)
C(21)	3594 (4)	3495 (3)	1362 (1)
C(22)	5974 (4)	3821 (3)	1366 (1)
C(23)	4728 (3)	4089 (3)	1161 (1)
C(24)	4581 (5)	5415 (4)	1009 (1)
C(25)	4666 (5)	4380 (3)	686 (1)
H(2)	207 (3)	182 (3)	188 (1)
H(3)	228 (3)	29 (3)	240 (1)
H(5)	327 (3)	−147 (3)	268 (1)
H(6)	492 (4)	−293 (4)	264 (1)
H(7)	651 (3)	−287 (3)	206 (1)
H(8)	634 (3)	−134 (3)	156 (1)
H(13)	415 (3)	−138 (3)	95 (1)
H(14)	419 (3)	−267 (3)	35 (1)
H(15)	571 (3)	−222 (3)	22 (1)
H(16)	717 (3)	−57 (3)	−15 (1)
H(18)	790 (3)	122 (3)	24 (1)
H(19)	766 (3)	260 (3)	81 (1)
H(211)	361 (3)	350 (3)	170 (1)
H(212)	286 (3)	392 (3)	126 (1)
H(221)	673 (3)	413 (3)	117 (1)
H(222)	605 (3)	423 (3)	166 (1)
H(241)	530 (3)	597 (3)	106 (1)
H(242)	371 (3)	578 (3)	105 (1)
H(251)	389 (3)	411 (3)	54 (1)
H(252)	544 (3)	430 (3)	53 (1)


(I)

Discussion. The molecule is shown in Fig. 1. The bond lengths and the valence angles are given in Fig. 2. The geometry of both naphthyl systems is very similar and is slightly distorted in comparison with that of naphthalene (Cruickshank, 1957). The angle of 71·5° between the naphthyl planes is smaller than the 76·6° found in 2,2′-dihydroxy-1,1′-binaphthalene-3,3′-

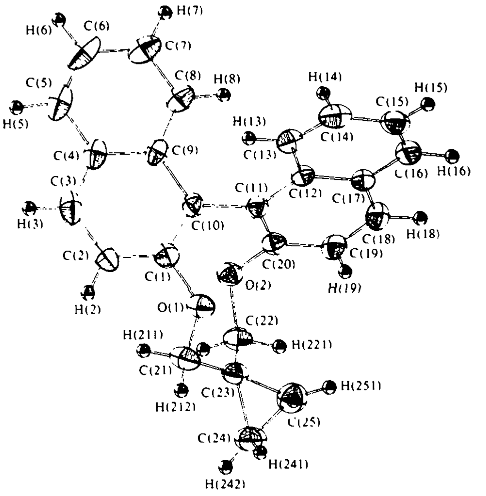


Fig. 1. Spiro[3,4-dihydro-2*H*-1,5-dioxadinaphtho[2,1-*f*:1,2-*h*]-cyclononene-3,1′-cyclopropane]. For non-hydrogen atoms the thermal ellipsoids are drawn at the 50% probability level (Johnson, 1965).

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* Lists of structure factors and thermal parameters are available from the author on request and have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34580 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

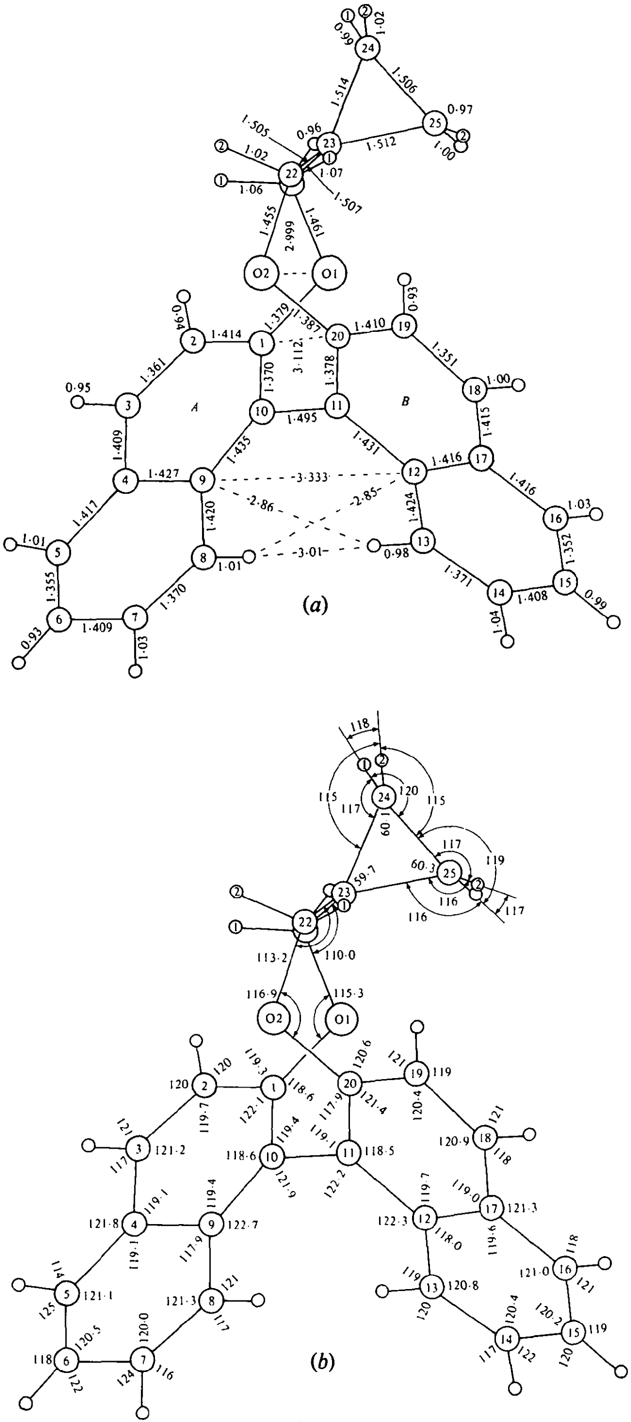

Fig. 2. (*a*) Bond lengths (Å) with some non-bonding intramolecular distances marked with broken lines; mean e.s.d.'s are 0·005 Å for C–C, C–O bonds and 0·03 Å for C–H bonds. (*b*) Valence angles (°) with mean e.s.d.'s of 0·3° for CCC, CCO angles and 2° for CCH, OCH, HCH angles. Missing angles are: C(21)C(23)C(22) 117·0, C(21)C(23)C(24) 116·6, C(21)C(23)-C(25) 117·3, C(22)C(23)C(24) 113·9, C(22)C(23)C(25) 119·6, C(23)C(21)H(211) 114, C(23)C(21)H(212) 108, O(1)C(21)-H(211) 109, O(1)C(21)H(222) 105, H(211)C(21)H(212) 110, C(23)C(22)H(221) 111, C(23)C(22)H(222) 112, O(2)C(22)-H(221) 109, O(2)C(22)H(222) 102, H(221)C(22)H(222) 109°.

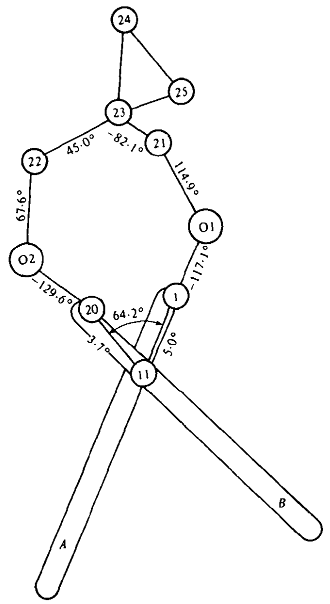

Fig. 3. Torsion angles (°) in the nine-membered ring viewed along the C(11)–C(10) bond. Standard deviations in the torsion angles are approximately 0·8°.

dicarboxylic acid dimethyl ester (Akimoto & Iitaka, 1969). The equations of the naphthyl best planes are, for *A*: $0\cdot5753x + 0\cdot6018y + 0\cdot5539z = 5\cdot6837$ with r.m.s.d. = 0·008 Å; for *B*: $0\cdot6725x - 0\cdot5606y + 0\cdot4833z = 5\cdot2411$ with r.m.s.d. = 0·051 Å. Some shorter intramolecular distances between the *A* and *B* naphthyl systems are marked in Fig. 2. The conformation of the nine-membered ring is described by the torsion angles given in Fig. 3. It is quite different from all the symmetric and asymmetric conformations considered for 1,3-cyclononadiene by Zuccarello, Buemi & Favini (1971). The C(1)C(10)C(11)C(20) torsion angle is not the same as that between the naphthyl planes, but this is due to strain in the C(10)–C(11) bond in the binaphthyl system: the deviation of C(11) from plane *A* is significant (+0·081 Å). The cyclopropane ring is symmetric within experimental error.

The shortest intermolecular distances C···H, O···H, and H···H are respectively 2·78 [C(5), H(2)], 2·84 [O(1), H(242)] and 2·30 Å [H(222), H(5)].

It is interesting to note that although on simple grounds it might be expected that the molecule should have a twofold axis, it very clearly does not (Fig. 2). Probably the very severe distortion from twofold symmetry results initially from steric hindrance between H(8) and H(13), the effect of which is transmitted through the molecule *via* the nine-membered ring to the cyclopropane group, causing it to bend strongly away from the expected twofold axis.

Intensity measurements were carried out in the X-ray Laboratory of ŚLAFiBS, Kraków.

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Structure of Spiro[3,4-dihydro-2*H*-1,5-dioxadinaphtho[2,1-*f*:1,2-*h*]cyclononene-3,1'-cyclopentane]

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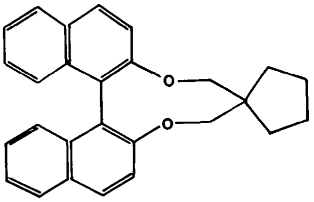
Abstract. $C_{27}H_{24}O_2$, M_r = 380·5, tetragonal, crystal faces: {101}, {101}, {100}, space group $P4_1(P4_3)$, a = 11·243 (5), c = 16·346 (6) Å, V = 2066 Å^3, Z = 4, D_x = 1·184 Mg m^{-3}, μ(Cu $K\alpha$) = 0·515 mm^{-1}. The structure was solved by direct methods and refined by full-matrix least squares to a final R value of 0·054 for 1328 reflections. The molecule is asymmetric. There is little influence of strain in the small ring on the conformation of the nine-membered ring when compared with the cyclopropane counterpart.

Introduction. The structure of spiro[3,4-dihydro-2*H*-1,5-dioxadinaphtho[2,1-*f*:1,2-*h*]cyclononene-3,1'-cyclopentane] (I) (SNPE) was investigated in order to compare it with that of spiro[3,4-dihydro-2*H*-1,5-dioxadinaphtho[2,1-*f*:1,2-*h*]cyclononene-3,1'-cyclopropane] (SNPR) (Stadnicka, 1979) and to find any effect of the strain in the small ring on the conformation of the dioxacyclononene ring. The effects of the conjugated systems can be analysed by comparison of the SNPE and SNPR structures with the structure of spiro[3,4-dihydro-2*H*-1,5-dioxadibenzo[*f*,*h*]-cyclononene-3,1'-cyclopropane] (SBPR) (Grochowski & Karle, 1978).



(I)

Transparent crystals of SNPE were synthesized by Smoliński & Paluchowska (1979). A crystal of approximate dimensions 0·2 × 0·2 × 0·3 mm was used to collect intensity data up to θ = 55° using an Enraf–Nonius CAD-4 diffractometer and graphite-monochromated Cu $K\alpha$ radiation in the $\omega/2\theta$ scan mode and (0·7 + 0·2 tan θ)° scan range. The unit-cell parameters were obtained by least-squares refinement from the setting angles of 15 reflections used in the auto-indexing procedure. The fluctuation of intensity for two standard reflections was less than 5%. The intensities were corrected for geometrical factors but not for absorption. Of the 2765 reflections measured, 1336 unique values were obtained in a sort–merge procedure giving an internal R = 0·028. Eight reflections with $|F_o| \leq \sigma(F_o)$ were treated as unobserved.

The structure was solved using the tangent formula on six selected initial phases. The E maps were calculated from 244 reflections (E > 1·2) and the solution with the best figure of merit revealed the positions of 27 non-hydrogen atoms. The two missing atoms of the cyclopentane ring were found in a Fourier difference map. Anisotropic full-matrix least-squares refinement and subsequent difference maps yielded positions of all the H atoms except H(221), H(241) and H(252), which were calculated instead. All H atoms were included in the refinement with isotropic thermal factors. The calculations were terminated when all the shifts were less than 0·2 of the e.s.d. and the discrepancy factors defined as $R = \sum|\Delta F|/\sum|F_o|$, $R_w = \sum w^{1/2}|\Delta F|/\sum w^{1/2}|F_o|$ and $R_G = [\sum w(\Delta F)^2/\sum wF_o^2]^{1/2}$ were 0·054, 0·042 and 0·038 respectively. The goodness-of-fit parameter S was 0·69. The peaks on the final Fourier difference map were less than 0·15 e Å^{-3}. The weighting scheme was $w = [\sigma(F_o)]^{-1}$ where $\sigma(F_o)$ is the standard deviation in the observed $|F(hkl)|$ values derived from counting statistics. The final

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