

Fig. 1. One molecule of diphenylfuroxan. The bond lengths shown have e.s.d.'s of 0.005 Å. O(22) is the alternative position found for  $\frac{1}{3}$  of the O(2) atoms.

Chiari & Viterbo, 1973), and two forms of methyl-*p*-bromophenylfuroxan (Calleri, Ferraris & Viterbo, 1969*a,b*). The atoms of the furoxan ring [N(1),N(2),O(1),O(2),C(1),C(8)] together with C(2) and C(9) are coplanar within 0.03 Å but the phenyl rings are twisted out of this plane; ring (I) is 17° from the furoxan plane and ring (II) 59°. Repulsion of non-bonded atoms would be expected to prevent the two rings being coplanar (as in stilbene or hexaphenylbenzene). In this crystal environment one phenyl ring is twisted much further out of plane than the other; thus

the alternative position, O(22), for the exocyclic O(2) corresponds to a stereochemically different isomer. Rapid interchange between isomers should be possible in solution. The conformation is similar to that of diphenylfuroxan (Mellini & Merlino, 1976) where the phenyl groups are twisted 19 and 61° from the furazan plane.

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#### References

- BOYER, J. H., REINISCH, R. F., DANZIG, M. J., STONER, G. A. & SAHAR, F. (1955). *J. Am. Chem. Soc.* **77**, 5688–5690.
- CALLERI, M., CHIARI, G. & VITERBO, D. (1973). *Cryst. Struct. Commun.* **2**, 335–338.
- CALLERI, M., FERRARIS, G. & VITERBO, D. (1969*a*). *Acta Cryst.* **B25**, 1126–1133.
- CALLERI, M., FERRARIS, G. & VITERBO, D. (1969*b*). *Acta Cryst.* **B25**, 1133–1139.
- MELLINI, M. & MERLINO, S. (1976). *Acta Cryst.* **B32**, 1079–1082.
- STEWART, J. M., KRUGER, G. J., AMMON, H. L., DICKINSON, C. & HALL, S. R. (1972). XRAY system – version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland; implemented for the ICL 4/75 computer at Edinburgh Regional Computing Centre.

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### (3*aRS*,6*RS*,6*aRS*,7*RS*,9*aSR*)-7-Benzyl-5,6-dimethyl-3,3*a*,6,6*a*,7,8-hexahydrofuro-[3,4-*h*]isoindole-1,9(9*aH*)-dione

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(Received 13 January 1978; accepted 28 January 1978)

**Abstract.** C<sub>19</sub>H<sub>21</sub>NO<sub>3</sub>, *M<sub>r</sub>* = 311.15, triclinic, *P* $\bar{1}$ , *a* = 11.128 (6), *b* = 12.016 (8), *c* = 13.722 (7) Å,  $\alpha$  = 113.37 (5),  $\beta$  = 94.45 (5),  $\gamma$  = 95.68 (6)°, *U* = 1622 Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.243 g cm<sup>-3</sup>,  $\mu$ (Mo *K*α) = 0.5 cm<sup>-1</sup>. There are two independent molecules, linked by hydrogen bonds, in the asymmetric unit. The structure was refined to *R* = 0.061 for 4209 independent reflexions. The relative configurations at the five asym-

metric centres have been established to be those given in the title.

**Introduction.** The title compound (Fig. 1) was prepared as an intermediate during studies towards the synthesis of cytochalasan (Owens & Raphael, 1978). The structure determination was undertaken to establish the relative configurations at the five asymmetric centres. A related compound, obtained in the same synthetic pathway, has also been investigated crystallo-

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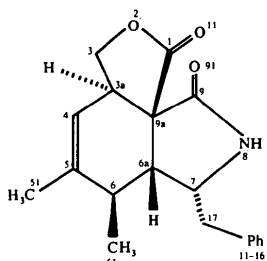


Fig. 1. The *RRRRS* isomer of the title compound. The atomic-numbering scheme for non-hydrogen atoms is shown [for the ring atoms C(1) to C(9a) this is the IUPAC numbering].

graphically (Jones & Kennard, 1978). Large colourless tabular crystals were obtained from diethyl ether/petroleum spirit. Intensities were measured on a Syntex  $P2_1$  four-circle diffractometer with Mo  $K\alpha$  radiation and a graphite monochromator, with a crystal  $0.9 \times 0.5 \times 0.35$  mm. Cell dimensions were obtained by least squares from 15 strong reflexions. The cell used was both the Dirichlet and Delaunay cell; no cell of higher symmetry could be found with a variety of lattice-geometry programs. 6468 reflexions were collected in the range  $0 < 2\theta < 50^\circ$ ; averaging equivalent reflexions

Table 1. *Atom coordinates* ( $\times 10^4$ ) *and isotropic temperature factors* ( $\text{\AA}^2 \times 10^3$ )

Anisotropic thermal parameters for C, N and O atoms have been deposited.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i>		<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i>
<b>Molecule A</b>					<b>Molecule B</b>				
C(1)	4096 (3)	7576 (3)	3055 (3)		C(1)	7419 (3)	-2111 (3)	-438 (2)	
O(2)	4542 (2)	8494 (3)	3986 (3)		O(2)	6359 (2)	-2732 (2)	-461 (2)	
C(3)	3670 (6)	8699 (4)	4715 (4)		C(3)	6079 (3)	-2494 (3)	606 (3)	
C(3a)	2599 (3)	7710 (3)	4244 (2)		C(3a)	6954 (2)	-1376 (3)	1369 (2)	
C(4)	2415 (3)	6913 (3)	4832 (2)		C(4)	6351 (3)	-244 (3)	1803 (2)	
C(5)	2501 (3)	5743 (3)	4455 (2)		C(5)	6632 (3)	774 (3)	1667 (2)	
C(6)	2873 (3)	5052 (3)	3358 (2)		C(6)	7578 (3)	895 (3)	976 (2)	
C(6a)	2585 (2)	5642 (2)	2580 (2)		C(6a)	8497 (2)	-8 (2)	819 (2)	
C(7)	1274 (2)	5312 (2)	1972 (2)		C(7)	9625 (2)	349 (2)	1674 (2)	
N(8)	1088 (2)	6426 (2)	1840 (2)		N(8)	9953 (2)	-849 (2)	1522 (2)	
C(9)	1925 (2)	7385 (3)	2374 (2)		C(9)	9072 (2)	-1790 (2)	989 (2)	
C(9a)	2815 (2)	7046 (2)	3076 (2)		C(9a)	7966 (2)	-1287 (2)	691 (2)	
C(11)	-994 (2)	4821 (3)	1981 (2)		C(11)	10563 (3)	1307 (3)	3627 (2)	
C(12)	-1806 (3)	5551 (3)	2545 (3)		C(16)	11728 (3)	1596 (4)	3441 (3)	
C(13)	-2970 (3)	5474 (4)	2072 (4)		C(15)	12711 (3)	1779 (4)	4187 (4)	
C(14)	-3334 (3)	4678 (4)	1037 (4)		C(14)	12560 (4)	1706 (4)	5135 (4)	
C(15)	-2543 (3)	3971 (3)	473 (3)		C(13)	11431 (4)	1427 (5)	5324 (3)	
C(16)	-1382 (3)	4010 (3)	930 (3)		C(12)	10433 (3)	1215 (4)	4577 (3)	
C(17)	273 (3)	4928 (4)	2525 (3)		C(17)	9457 (3)	1103 (3)	2833 (2)	
C(51)	2259 (6)	5016 (4)	5114 (3)		C(51)	5957 (4)	1865 (4)	2142 (3)	
C(61)	4220 (3)	4904 (4)	3464 (3)		C(61)	6943 (3)	743 (3)	-123 (3)	
O(11)	4683 (2)	7259 (3)	2332 (3)		O(11)	7857 (2)	-2235 (2)	-1241 (2)	
O(91)	1982 (2)	8406 (2)	2365 (2)		O(91)	9113 (2)	-2876 (2)	777 (2)	
H(1)	432 (31)	6510 (31)	1488 (26)	89 (3)	H(1)	10632 (31)	-992 (31)	1768 (26)	89 (3)
H(2)	1218 (25)	4609 (25)	1284 (21)	70 (2)	H(2)	10279 (26)	819 (26)	1489 (22)	70 (2)
H(3)	3120 (26)	5404 (26)	2031 (23)	70 (2)	H(3)	8747 (26)	-179 (25)	125 (24)	70 (2)
H(4)	2390 (26)	4198 (28)	3027 (23)	70 (2)	H(4)	8026 (26)	1755 (27)	1281 (22)	70 (2)
H(5)	2140 (30)	7272 (30)	5519 (28)	89 (3)	H(5)	5730 (30)	-278 (29)	2288 (25)	89 (3)
H(6)	1883 (27)	8119 (27)	4238 (22)	70 (2)	H(6)	7286 (26)	-1570 (26)	1975 (23)	70 (2)
H(7)	270 (26)	5514 (28)	3262 (25)	70 (2)	H(7)	8693 (27)	628 (26)	3007 (22)	70 (2)
H(8)	421 (27)	4232 (30)	2553 (24)	70 (2)	H(8)	9286 (26)	1937 (28)	2882 (22)	70 (2)
H(9)	-1507 (29)	6160 (30)	3327 (27)	89 (3)	H(13)	11931 (31)	1703 (32)	2867 (28)	89 (3)
H(10)	-3574 (31)	5926 (30)	2480 (26)	89 (3)	H(12)	13448 (32)	1944 (31)	3968 (27)	89 (3)
H(11)	-4044 (33)	4742 (31)	797 (27)	89 (3)	H(11)	13255 (31)	1896 (30)	5666 (27)	89 (3)
H(12)	-2673 (32)	3524 (33)	-161 (28)	89 (3)	H(10)	11241 (30)	1295 (30)	5934 (28)	89 (3)
H(13)	-775 (31)	3504 (30)	578 (26)	89 (3)	H(9)	9566 (31)	906 (30)	4698 (25)	89 (3)
H(14)	4073	8694	5455	70 (2)	H(14)	5251 (29)	-2378 (26)	649 (22)	70 (2)
H(15)	3372	9575	4871	70 (2)	H(15)	6278 (27)	-3127 (30)	724 (24)	70 (2)
H(16)	4443	4373	2675	102 (4)	H(16)	7617	871	-609	102 (4)
H(17)	4384	4429	3974	102 (4)	H(17)	6330	1415	-2	102 (4)
H(18)	4780	5789	3797	102 (4)	H(18)	6434	-165	-523	102 (4)
H(19)	2400	4080	4670	102 (4)	H(19)	6320	2597	1942	102 (4)
H(19')	1530	4274	4682	102 (4)	H(19')	6576	2650	2690	102 (4)
H(20)	1327	5034	5283	102 (4)	H(20)	6039	2178	3007	102 (4)
H(20')	1995	5600	5871	102 (4)	H(20')	5248	1653	2577	102 (4)
H(21)	2865	5407	5858	102 (4)	H(21)	4992	1600	1830	102 (4)
H(21')	3068	4647	5257	102 (4)	H(21')	5528	2072	1512	102 (4)

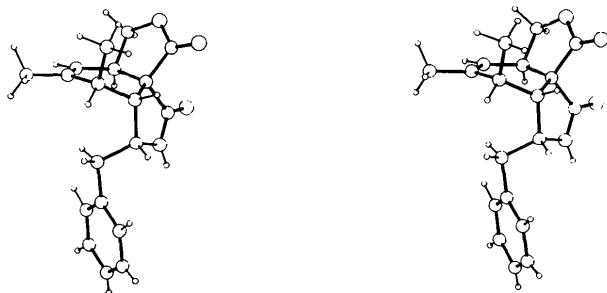
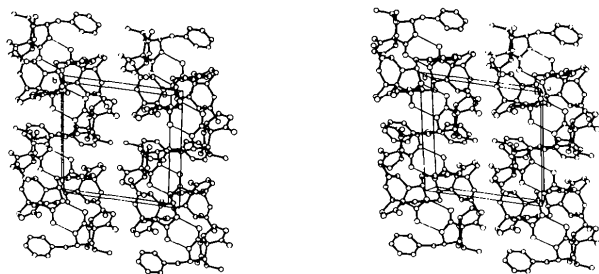
Fig. 2. Stereoview of molecule *A*.

Fig. 3. Stereo packing diagram showing the hydrogen bonding (thin lines). H atoms are omitted.

Table 2. Bond lengths (Å)

	Molecule <i>A</i>	Molecule <i>B</i>
C(9)—O(91)	1.227 (5)	1.225 (5)
C(9)—N(8)	1.321 (4)	1.337 (4)
C(9)—C(9a)	1.522 (6)	1.521 (6)
C(7)—N(8)	1.451 (5)	1.458 (5)
C(7)—C(6a)	1.554 (5)	1.550 (6)
C(7)—C(17)	1.529 (7)	1.524 (6)
C(6a)—C(6)	1.529 (7)	1.529 (6)
C(6a)—C(9a)	1.534 (6)	1.527 (6)
C(6)—C(5)	1.516 (6)	1.503 (7)
C(6)—C(61)	1.530 (7)	1.548 (7)
C(5)—C(4)	1.307 (6)	1.320 (6)
C(5)—C(51)	1.508 (9)	1.517 (7)
C(4)—C(3a)	1.487 (7)	1.502 (6)
C(3a)—C(3)	1.497 (7)	1.522 (6)
C(3a)—C(9a)	1.531 (6)	1.538 (6)
C(11)—C(17)	1.512 (6)	1.511 (6)
C(11)—C(12)	1.383 (5)	1.368 (6)
C(11)—C(16)	1.387 (5)	1.378 (6)
C(12)—C(13)	1.380 (6)	1.382 (7)
C(13)—C(14)	1.361 (6)	1.339 (8)
C(14)—C(15)	1.352 (6)	1.360 (9)
C(15)—C(16)	1.380 (6)	1.376 (7)
C(3)—O(2)	1.416 (8)	1.441 (6)
C(1)—O(2)	1.330 (5)	1.323 (5)
C(1)—O(11)	1.186 (6)	1.201 (5)
C(9a)—C(1)	1.510 (6)	1.505 (5)

(Lp corrections being applied) gave 4218 reflexions with  $F > 4\sigma(F)$ .

The structure was solved by multiresolution  $\Sigma_2$  sign expansion with 546  $E$  values  $>1.2$  which gave sites for all non-hydrogen atoms. Isotropic least-squares refine-

Table 3. Bond angles ( $^\circ$ )

	Molecule <i>A</i>	Molecule <i>B</i>
O(91)—C(9)—N(8)	127.6 (4)	127.4 (4)
O(91)—C(9)—C(9a)	123.9 (3)	124.5 (3)
N(8)—C(9)—C(9a)	108.4 (4)	108.1 (3)
C(9)—N(8)—C(7)	115.8 (3)	114.8 (3)
N(8)—C(7)—C(6a)	102.5 (3)	101.5 (3)
N(8)—C(7)—C(17)	111.8 (3)	111.9 (4)
C(6a)—C(7)—C(17)	116.1 (4)	117.4 (3)
C(7)—C(6a)—C(6)	117.4 (3)	118.4 (3)
C(7)—C(6a)—C(9a)	104.5 (3)	104.1 (3)
C(6)—C(6a)—C(9a)	114.8 (3)	115.1 (3)
C(6a)—C(6)—C(5)	112.8 (4)	113.5 (4)
C(6a)—C(6)—C(61)	111.6 (4)	110.1 (3)
C(5)—C(6)—C(61)	110.0 (4)	109.6 (4)
C(6)—C(5)—C(4)	122.6 (4)	122.3 (4)
C(6)—C(5)—C(51)	116.4 (4)	115.9 (4)
C(4)—C(5)—C(51)	120.9 (4)	121.6 (4)
C(5)—C(4)—C(3a)	125.5 (4)	126.2 (4)
C(4)—C(3a)—C(3)	114.4 (4)	112.7 (3)
C(4)—C(3a)—C(9a)	115.1 (3)	113.8 (4)
C(3)—C(3a)—C(9a)	103.5 (4)	103.2 (3)
C(7)—C(17)—C(11)	113.0 (4)	114.0 (3)
C(17)—C(11)—C(12)	119.4 (3)	120.2 (4)
C(17)—C(11)—C(16)	122.4 (4)	122.5 (4)
C(12)—C(11)—C(16)	118.1 (4)	117.3 (4)
C(11)—C(12)—C(13)	120.8 (4)	121.1 (5)
C(12)—C(13)—C(14)	120.3 (5)	121.1 (6)
C(13)—C(14)—C(15)	119.5 (5)	118.7 (5)
C(14)—C(15)—C(16)	121.6 (4)	121.1 (5)
C(11)—C(16)—C(15)	119.7 (4)	120.7 (5)
C(3a)—C(3)—O(2)	109.5 (4)	107.1 (4)
C(3)—O(2)—C(1)	110.4 (4)	111.2 (3)
O(2)—C(1)—O(11)	121.1 (4)	121.8 (3)
O(2)—C(1)—C(9a)	111.5 (4)	111.5 (4)
O(11)—C(1)—C(9a)	127.4 (4)	126.6 (4)
C(9)—C(9a)—C(6a)	103.4 (3)	103.1 (3)
C(9)—C(9a)—C(3a)	108.5 (3)	110.2 (4)
C(6a)—C(9a)—C(3a)	117.6 (4)	117.3 (3)
C(9)—C(9a)—C(1)	109.5 (4)	109.0 (3)
C(6a)—C(9a)—C(1)	113.9 (3)	113.3 (4)
C(3a)—C(9a)—C(1)	103.8 (3)	104.0 (3)

ment gave  $R = 0.19$ . Anisotropic refinement ( $R = 0.12$ ), followed by a difference synthesis, revealed 29 of the 42 H atoms in the asymmetric unit. In the final stages of refinement all H atoms except methyl H and the H on C(3), molecule *A* (which possessed high thermal parameters), were allowed to refine freely starting from calculated positions. Methyl groups attached to  $sp^3$  C were refined as rigid groups with C—H 1.08 Å, H—C—H 109.5°; these constraints were also applied to the H on C(3), molecule *A*. Methyl groups on  $sp^2$  C refined successfully with a model of twofold disorder (Jones & Kennard, 1977); site-occupation factors refined to 0.32 [H(19) to H(21)], 0.68 [H(19') to H(21')] (molecule *A*) and 0.27 [H(19) to H(21)], 0.73 [H(19') to H(21')] (molecule *B*) (all e.s.d.'s 0.01). Chemically similar H atoms were assigned equal isotropic temperature factors. Nine reflexions considerably in error were omitted. The final  $R' = \sum w^{1/2} \Delta / \sum w^{1/2} |F_o|$  was 0.072, with a corre-

sponding  $R$  of 0.061.\* The weighting scheme was  $w = 1/[\sigma^2(F) + 0.001F^2]$ , which gave a mean  $w\Delta^2$  varying only slightly with  $\sin \theta$  or  $|F_o|$ . A final difference map had no peaks  $>0.26 \text{ e } \text{\AA}^{-3}$ .

Final atomic coordinates are given in Table 1, and bond distances and angles in Tables 2 and 3. Diagrams of the structure are given in Figs. 2 and 3.

**Discussion.** The stereochemistry at the asymmetric centres is established as (3a*RS*,6*RS*,6a*RS*,7*RS*,9a*SR*). The chemical significance of this is discussed elsewhere (Owens & Raphael, 1978). The two molecules in the asymmetric unit are very similar; the only bond-length differences  $>0.02 \text{ \AA}$  are for C(3a)–C(3) and C(3)–O(2), and the only bond-angle difference  $>2^\circ$  is for C(3a)–C(3)–O(2). There are several small torsion-angle differences (up to  $8^\circ$ ) but the major difference is in the orientation of the benzene rings, C(12)–C(11)–C(17)–C(7) being  $-119.6$ ,  $-137.9^\circ$  and C(16)–C(11)–C(17)–C(7)  $60.6$ ,  $42.4^\circ$  in molecules *A* and *B* respectively. The angles C(5)–C(4)–C(3a) and O(11)–C(1)–C(9a) are considerably greater than  $120^\circ$  in both molecules, which may be due to ring strain.

\* Lists of structure factors, anisotropic thermal parameters and bond distances and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33374 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

In the cyclohexene rings, C(3a) lies  $0.56$ ,  $0.47 \text{ \AA}$  above and C(7a)  $0.17$ ,  $0.04 \text{ \AA}$  above the plane of the other four atoms in molecules *A* and *B* respectively. This corresponds to a flattened form of the sofa conformation (Bucourt & Hainaut, 1965).

The crystallographically independent molecules are linked in pairs by two hydrogen bonds connecting the N–H and O=C of the isoindole moieties (Fig. 3). Relevant distances ( $\text{\AA}$ ) are O(91)⋯N(8)  $2.92$ , O(91)⋯H(1)  $2.05$  and N(8)⋯O(91)  $2.84$ , H(1)⋯O(91)  $1.99 \text{ \AA}$  (first atoms in molecule *B* at  $x, y, z$ ; second atoms in molecule *A* at  $1 + x, -1 + y, z$ ).

We thank the MRC for financial support and the SRC for provision of the diffractometer. Figs. 2 and 3 were drawn with *PLUTO* written by Dr W. D. S. Motherwell. All other crystallographic programs were written by Dr G. M. Sheldrick.

#### References

- BUCOURT, R. & HAINAUT, D. (1965). *Bull. Soc. Chim. Fr.* pp. 1366–1378.  
 JONES, P. G. & KENNARD, O. (1977). *Acta Cryst.* B33, 627–630.  
 JONES, P. G. & KENNARD, O. (1978). *Acta Cryst.* B34, 2025–2027.  
 OWENS, C. & RAPHAEL, R. A. (1978). *J. Chem. Soc. Perkin Trans.* 1. In the press.

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### (3a*SR*,4*RS*,5*SR*,7a*SR*)-4-(1-Oxo-2-phenylethyl)-5,6-dimethyl-1,4,5,7a-tetrahydroisobenzofuran-3(3a*H*)-one

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**Abstract.**  $\text{C}_{18}\text{H}_{20}\text{O}_3$ ,  $M_r = 285.36$ , monoclinic,  $P2_1/c$ ,  $a = 12.703(4)$ ,  $b = 11.754(4)$ ,  $c = 10.932(4) \text{ \AA}$ ,  $\beta = 109.34(2)^\circ$ ,  $U = 1540 \text{ \AA}^3$ ,  $Z = 4$ ,  $D_x = 1.230$ ,  $D_m$  (floatation)  $= 1.23 \text{ g cm}^{-3}$ ,  $\mu(\text{Cu } K\alpha) = 5.8 \text{ cm}^{-1}$ . The structure was refined to  $R = 0.056$  for 1862 unique reflexions. The relative configurations at the four asymmetric centres are established to be those given in the title.

**Introduction.** The title compound (Fig. 1) is a synthetic intermediate prepared during studies towards the synthesis of cytochalasan (Owens & Raphael, 1978). The structure determination was undertaken to establish the relative configurations at the four asymmetric centres. A related compound, obtained in the same synthetic pathway, has also been investigated crystallographically (Jones & Kennard, 1978).

Colourless crystals were obtained from diethyl ether in two forms. The first form, approximately equi-

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