Acta Cryst. (1974). B**30**, 338

(±)-2,2′-Spirobi[indan]-1,1′-dione

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(*Received* 7 *August* 1973; *accepted* 8 *August* 1973)

$C_{17}H_{12}O$, triclinic, $P\bar{1}$, $a=9{\cdot}375$ (12), $b=7{\cdot}898$ (7), $c=9{\cdot}139$ (11) Å, $\alpha=74{\cdot}14$ (9), $\beta=90{\cdot}86$ (9), $\gamma=108{\cdot}30$ (7)°, $Z=2$, $d_c=1{\cdot}268$, $d_m=1{\cdot}31$ g cm³. The structure was solved by the multisolution method and refined to $R=0{\cdot}08$.

Introduction

The compound was supplied by Dynesen (1972) and the colourless crystals were made by zone melting. Intensities were collected on a four-circle Picker diffractometer. Cu $K\alpha$ radiation ($\lambda=1{\cdot}5418$ Å) was selected by a graphite monochromator. The counting chain included a scintillation counter and a pulse-height analyser. Reflexions were collected for a full sphere up to $\sin\theta/\lambda=0{\cdot}600$ Å^{-1}. Because of absorption in the mounting material the intensities of reflexions related by symmetry differed significantly. Lorentz and polarization corrections were applied, assuming graphite to behave as an ideal mosaic crystal. From each symmetry-related pair of reflexions the larger was chosen for inclusion in the data set. This gave 2072 F values, which were converted to E values by the program *SYMBAD* (Danielsen, 1969). The structure was solved by direct methods with the program *MULTAN* (Main, Woolfson & Germain, 1971). The 454 largest E values were used. A Fourier map was calculated with the set of phases which had the best figure of merit after tangent refinement. It showed all the non-hydrogen atoms. Full matrix least-squares refinement was carried out for the positional and anisotropic temperature factor parameters of the non-hydrogen atoms. Positional parameters of hydrogen atoms were derived from stereochemical considerations and were included in the least-squares computations with isotropic temperature factor parameters. Convergence was obtained at $R=0{\cdot}096$ ($R=\sum||F_{\rm obs}|-|F_c||/\sum|F_{\rm obs}|$) with all data included. When intensities with $F^2\le 2\sigma(F^2)$ were excluded an R of 0·08 was obtained. A difference map calculated after the last refinement with the significant F values showed no features which could be interpreted as atoms. Final values of the atomic coordinates and thermal parameters are listed in Table 1. Bond lengths are given in

Table 1. *Atomic coordinates and thermal parameters* (× 10³)

The anisotropic temperature parameters of the non-hydrogen atoms are of the form

$$\exp\left[-2\pi^2(u_{11}a_1^{*2}h^2+u_{22}a_2^{*2}k^2+u_{33}a_3^{*2}l^2+u_{12}a_1^*a_2^*hk+u_{23}a_2^*a_3^*kl+u_{13}a_1^*a_3^*hl)\right],$$

a_i^* being the size of the ith reciprocal axis in Å^{-1}. The isotropic temperature parameters of the hydrogen atoms are of the form $\exp[-2\pi^2U\sin^2\theta/\lambda^2]$. U and u_{ij} are in Å².

(*a*) Non-hydrogen atoms

	x	y	z	u_{11}	u_{22}	u_{33}	u_{12}	u_{13}	u_{23}
O	0·4000 (3)	0·6130 (3)	0·8207 (3)	50 (1)	53 (1)	84 (2)	6 (1)	−1 (1)	−13 (1)
O′	0·1844 (3)	0·2954 (3)	0·5833 (3)	100 (2)	45 (1)	89 (2)	15 (1)	4 (2)	−15 (1)
C(1)	0·2690 (3)	0·5238 (4)	0·8196 (4)	48 (2)	43 (2)	67 (2)	11 (1)	3 (1)	−14 (1)
C′(1)	0·2660 (4)	0·4614 (5)	0·5685 (4)	57 (2)	47 (2)	71 (2)	13 (2)	−9 (2)	−14 (2)
C(2)	0·1748 (4)	0·5535 (4)	0·6793 (4)	53 (2)	46 (2)	61 (2)	11 (1)	4 (1)	−8 (1)
C(3)	0·0107 (4)	0·4480 (6)	0·7453 (5)	49 (2)	64 (2)	74 (2)	13 (2)	−2 (2)	−15 (2)
C′(3)	0·2176 (4)	0·7605 (5)	0·5874 (4)	63 (2)	45 (2)	69 (2)	18 (2)	1 (2)	−11 (2)
C(4)	0·0228 (4)	0·3306 (4)	0·9019 (4)	50 (2)	45 (2)	71 (2)	10 (2)	6 (2)	−18 (2)
C′(4)	0·3188 (4)	0·7810 (4)	0·4531 (4)	53 (2)	49 (2)	59 (2)	15 (1)	−5 (1)	−11 (2)
C(5)	0·9102 (4)	0·1917 (5)	0·0026 (5)	56 (2)	53 (2)	87 (3)	8 (2)	16 (2)	−20 (2)
C′(5)	0·3989 (4)	0·9420 (5)	0·3420 (5)	62 (2)	50 (2)	74 (2)	94 (2)	1 (2)	−9 (2)
C(6)	0·9475 (5)	0·1007 (5)	0·1417 (4)	84 (3)	49 (2)	85 (3)	11 (2)	32 (2)	−13 (2)
C′(6)	0·4788 (5)	0·9262 (6)	0·2237 (5)	66 (2)	68 (2)	66 (2)	10 (2)	3 (2)	−7 (2)
C(7)	0·0949 (5)	0·1454 (6)	0·1818 (5)	95 (3)	61 (2)	68 (2)	28 (2)	12 (2)	−8 (2)
C′(7)	0·4822 (5)	0·7539 (6)	0·2146 (5)	72 (2)	81 (3)	68 (2)	25 (2)	6 (2)	−19 (2)
C(8)	0·2079 (5)	0·2844 (5)	0·0837 (4)	68 (2)	62 (2)	65 (2)	23 (2)	−3 (2)	−20 (2)
C′(8)	0·4041 (4)	0·5951 (6)	0·3250 (4)	67 (2)	62 (2)	68 (2)	21 (2)	−4 (2)	−22 (2)
C(9)	0·1699 (4)	0·3746 (4)	0·9431 (4)	54 (2)	45 (2)	64 (2)	13 (1)	2 (2)	−2 (2)
C′(9)	0·3225 (4)	0·6097 (4)	0·4435 (4)	53 (2)	49 (2)	61 (2)	14 (1)	−7 (2)	−13 (2)

Table 1 (*cont.*)

(*b*) Hydrogen atoms

	x	*y*	*z*	*U*
H(1)	0·9547 (40)	0·3708 (51)	0·6758 (42)	71 (11)
H′(1)	0·1225 (50)	0·7760 (58)	0·5547 (47)	89 (13)
H(2)	0·9602 (51)	0·5354 (62)	0·7578 (49)	96 (14)
H′(2)	0·2704 (44)	0·8442 (55)	0·6464 (43)	75 (11)
H(3)	0·8062 (42)	0·1590 (49)	0·9759 (40)	70 (9)
H′(3)	0·4030 (42)	0·0662 (58)	0·3597 (44)	77 (11)
H(4)	0·8668 (48)	0·9975 (60)	0·2048 (46)	89 (13)
H′(4)	0·5445 (41)	0·0475 (53)	0·1441 (42)	68 (10)
H(5)	0·1117 (51)	0·0826 (66)	0·2868 (57)	107 (15)
H′(5)	0·5407 (49)	0·7491 (58)	0·1270 (50)	87 (12)
H(6)	0·3060 (39)	0·3205 (45)	0·1055 (36)	57 (9)
H′(6)	0·3960 (52)	0·4835 (69)	0·3133 (52)	92 (14)

Table 2 and angles in Table 3. The molecular structure is shown in Fig. 1. Observed and calculated structure factors are listed in Table 4.

Discussion

This investigation was undertaken to obtain information about the interaction between the π system of the benzene ring and the dione moiety of the molecule. The bond lengths of one of the five-membered rings are therefore compared with the bond lengths of (*S*)-(−)-spiro[4,4]nonane-1,6-dione (Altona, de Graaff, Leeuwestein & Romers, 1971) in Table 5.

Table 2. *Bond lengths* (Å)

$\Delta l = |l[C(i)\text{–}C(j)] - l[C'(i)\text{–}C'(j)]|$.
(*ORFFE*, Busing, Martin & Levy, 1964).

	l		*l*	Δl (Å) × 10³
C(4)–C(5)	1·383 (7)	C′(4)–C′(5)	1·389 (5)	6 (7)
C(5)–C(6)	1·378 (6)	C′(5)–C′(6)	1·380 (6)	2 (9)
C(6)–C(7)	1·385 (7)	C′(6)–C′(7)	1·396 (6)	10 (9)
C(7)–C(8)	1·375 (6)	C′(7)–C′(8)	1·372 (6)	4 (8)
C(8)–C(9)	1·390 (5)	C′(8)–C′(9)	1·385 (6)	5 (8)
C(4)–C(9)	1·386 (5)	C′(4)–C′(9)	1·391 (5)	5 (7)
C(1)–C(2)	1·553 (5)	C′(1)–C(2)	1·557 (5)	4 (8)
C(2)–C(3)	1·537 (5)	C(2)–C′(3)	1·545 (5)	8 (7)
C(3)–C(4)	1·502 (6)	C′(3)–C′(4)	1·504 (6)	2 (8)
C(1)–C(9)	1·463 (6)	C′(1)–C′(9)	1·459 (6)	4 (6)
O—C(1)	1·210 (4)	O′—C′(1)	1·213 (4)	3 (6)
H(3)–C(5)	0·973 (36)	H′(3)–C′(5)	1·028 (40)	53 (55)
H(4)–C(6)	0·953 (42)	H′(4)–C′(6)	1·046 (43)	75 (54)
H(5)–C(7)	0·988 (48)	H′(5)–C′(7)	0·992 (46)	5 (64)
H(6)–C(8)	0·908 (32)	H′(6)–C′(8)	0·899 (50)	14 (59)
H(1)–C(3)	1·026 (37)	H′(1)–C′(3)	0·971 (39)	58 (58)
H(2)–C(3)	0·984 (48)	H′(2)–C′(3)	0·977 (46)	7 (56)

Table 3. *Bond angles* (°)

$\Delta v = |v[C(i)\text{–}C(j)\text{–}C(k)] - v[C'(i)\text{–}C'(j)\text{–}C'(k)]|$ (*ORFFE*, Busing, Martin & Levy, 1964).

	v(°)		v(°)	$\Delta v \times 10$
C(9)–C(1)–C(2)	107·4 (3)	C′(9)–C′(1)–C′(2)	107·8 (3)	4 (4)
C(1)–C(2)–C(3)	104·0 (3)	C′(1)–C′(2)–C′(3)	103·8 (3)	2 (5)
C(2)–C(3)–C(4)	105·1 (3)	C′(2)–C′(3)–C′(4)	105·0 (3)	1 (4)
C(3)–C(4)–C(9)	111·6 (3)	C′(3)–C′(4)–C′(9)	111·8 (3)	1 (4)
C(4)–C(9)–C(1)	109·8 (3)	C′(4)–C′(9)–C′(1)	109·7 (3)	1 (4)
C(4)–C(5)–C(6)	119·0 (4)	C′(4)–C′(5)–C′(6)	118·5 (4)	5 (5)
C(5)–C(6)–C(7)	121·3 (4)	C′(5)–C′(6)–C′(7)	121·7 (4)	4 (5)
C(6)–C(7)–C(8)	120·4 (4)	C′(6)–C′(7)–C′(8)	119·7 (4)	7 (6)
C(7)–C(8)–C(9)	118·0 (4)	C′(7)–C′(8)–C′(9)	118·9 (4)	9 (5)
C(8)–C(9)–C(4)	121·9 (3)	C′(8)–C′(9)–C′(4)	121·6 (3)	3 (4)
C(9)–C(4)–C(5)	119·4 (4)	C′(9)–C′(4)–C′(5)	119·6 (4)	2 (5)
C(9)–C(1)–O	128·7 (3)	C′(9)–C′(1)–O′	127·7 (4)	
C(2)–C(1)–O	124·0 (3)	C′(2)–C′(1)–O′	124·3 (3)	
C(3)–C(4)–C(5)	129·0 (4)	C′(3)–C′(4)–C′(5)	128·6 (3)	
C(1)–C(9)–C(8)	128·3 (3)	C′(1)–C′(9)–C′(8)	128·7 (3)	

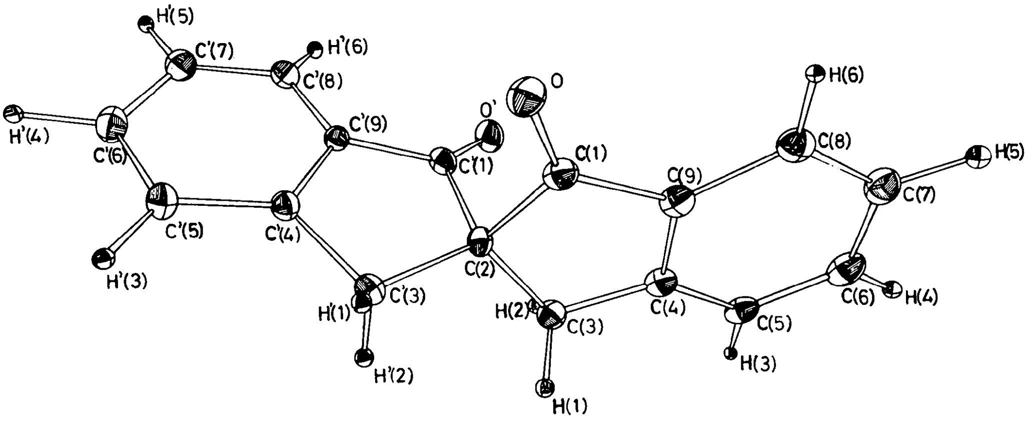

Fig. 1 The plane containing the atoms C(4), C(5) and C(9) is rotated 45° out of the plane of the paper. The thermal ellipsoids of the non-hydrogen atoms are scaled to enclose 75% probability (*ORTEP*, Johnson, 1965).

Table 4. *Observed and calculated structure factors*

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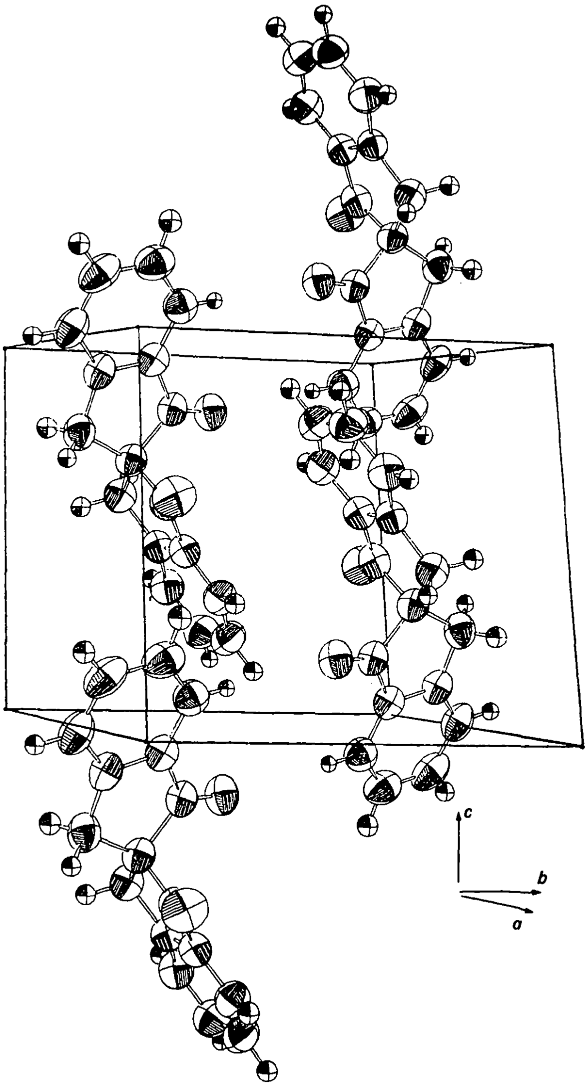

Fig. 2. The crystal structure of the title compound. The content of one unit cell is shown. The thermal ellipsoids of the non-hydrogen atoms are scaled to enclose 75% probability (*ORTEP*, Johnson, 1965).

The two halves of the latter molecule are related by crystallographic symmetry and the two halves of the title compound do not differ significantly as will be discussed later. Consequently only half the bond lengths of each of the two molecules need to be discussed. The first four bond lengths recorded for the title compound are significantly shorter than the corresponding bond lengths of the nonanedione. This means that the π-system of the benzene ring is interacting with the oxygen atom and the two nearest carbon atoms of the five-membered ring. The two remaining pairs of corresponding bond lengths do not differ significantly.

Table 5. *Bond lengths* (Å) *for one of the five-membered rings in the title compound and for* (*S*)-(−)-*spiro*[4,4]*nonane*-1,6-*dione*

The atoms of the latter compound are here labelled corresponding to the title compound.

	(*S*)-(−)-Spiro[4,4]-nonane-1,6-dione	(±)-2,2′-Spirobi[indan]-1,1′-dione
C(4)–C(9)	1·529 (5)	1·386 (5)
C(1)–C(9)	1·522 (5)	1·463 (6)
C(3)–C(4)	1·548 (5)	1·502 (6)
C(1)–O	1·239 (5)	1·210 (4)
C(2)–C(3)	1·538 (5)	1·537 (5)
C(1)–C(2)	1·529 (5)	1·553 (5)

Calculations of best least-squares planes through each of the benzene rings and the five-membered rings show that the benzene rings are planar within experimental error, whereas the five-membered rings are non-planar. Each of the latter is close to an envelope form.

The two halves of the molecule of the title compound are not related by crystallographic symmetry. The differences between corresponding bond lengths, $\Delta l = |l[C(i)\text{-}C(j)] - l[C'(i)\text{-}C'(j)]|$ and the standard deviations $\sigma(\Delta l)$ are recorded in Table 2. The differences between corresponding angles, $\Delta v = |v[C(i)\text{-}C(j)\text{-}C(k)] - v[C'(i)\text{-}C'(j)\text{-}C'(k)]|$ and the standard deviations $\sigma(\Delta v)$ are recorded in Table 3. The tables show that $\Delta l \leq 2\sigma(\Delta l)$ and $\Delta v \leq 2\sigma(\Delta v)$. Thus the hypothesis that the two halves of the molecule are equal cannot be rejected at the 5% significance level.

The crystal structure is shown in Fig. 2. The molecules form chains which twist in a manner such that opposite planes are always parallel.

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