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1-(α,α-Diphenylmethylene)-1,2-naphthoquinone*

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Abstract. $C_{23}H_{16}O$, $M_r = 308 \cdot 4$, monoclinic, $P2_1/c$ (C_{2h}^5, No. 14), $a = 9 \cdot 360$ (2), $b = 10 \cdot 183$ (2), $c = 17 \cdot 823$ (3) Å, $\beta = 109 \cdot 32$ (1)°, $Z = 4$, $D_c = 1 \cdot 28$ g cm^{-3}, μ(Mo $K\alpha$) = $0 \cdot 8$ cm^{-1}. The structure of the molecule shows considerable evidence of steric overcrowding at the exocyclic C=C double bond. In addition to a twist around this bond, there is a significant pyramidal distortion at the two sp^2 carbon atoms.

Introduction. The red-orange crystals (m.p. 199–200°C) of 1-(α,α-diphenylmethylene)-1,2-naphthoquinone (I) grow as long rods with well defined faces. The cell constants and intensity data out to $2\theta = 50°$ were obtained on a Picker FACS-1 diffractometer (Mo $K\alpha$, $\lambda = 0 \cdot 71069$ Å) using a crystal of dimensions $0 \cdot 5 \times 0 \cdot 3 \times 0 \cdot 3$ mm. 2435 out of the possible 3703 reflections were above the $2\sigma(I)$ significance level. The structure was determined by direct methods (Germain, Main & Woolfson, 1971), the H atoms were located from a difference map, and the refinement by full-

matrix least-squares methods gave final agreement factors of $R = 0 \cdot 060$ and $R_w = 0 \cdot 064$. The final value of $[\Sigma w(|F_o| - |F_c|)^2/(m - n)]^{1/2}$, where m is the number of observations and n is the number of variables, was $1 \cdot 79$. The atomic scattering factors were from the compilation in *International Tables for X-ray Crystallography* (1974). The atomic coordinates are listed in Table 1.†
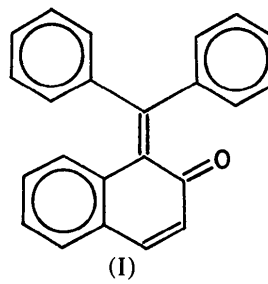
(I)

* *Chemical Abstracts* name: 1-Diphenylmethylene-2(1H)-naphthalenone.

Table 1. *Atomic coordinates for* (I)

Hydrogen atoms are given the number of the atom to which they are attached.

	x	y	z		z	y	z
C(1)	0·1529 (2)	0·0535 (2)	0·2395 (1)	C(21)	−0·1026 (3)	0·0184 (2)	0·3126 (1)
C(2)	0·0104 (2)	0·0840 (2)	0·1739 (1)	C(22)	0·0414 (2)	0·0717 (2)	0·3499 (1)
C(3)	−0·0376 (3)	0·0049 (3)	0·1066 (1)	C(23)	0·0684 (3)	0·1345 (2)	0·4236 (1)
C(4)	−0·1673 (3)	0·0347 (3)	0·0446 (1)	O	0·4349 (2)	0·0115 (2)	0·3693 (1)
C(5)	−0·2500 (3)	0·1442 (3)	0·0479 (2)	H(3)	0·016 (3)	−0·062 (2)	0·103 (1)
C(6)	−0·2027 (3)	0·2247 (3)	0·1127 (1)	H(4)	−0·199 (3)	−0·027 (2)	−0·001 (2)
C(7)	−0·0732 (3)	0·1960 (2)	0·1755 (1)	H(5)	−0·334 (3)	0·165 (2)	0·003 (1)
C(8)	0·2823 (2)	0·0267 (2)	0·2097 (1)	H(6)	−0·259 (3)	0·300 (3)	0·115 (1)
C(9)	0·3427 (3)	0·1299 (3)	0·1785 (1)	H(7)	−0·039 (2)	0·259 (2)	0·222 (1)
C(10)	0·4584 (3)	0·1056 (3)	0·1481 (2)	H(9)	0·300 (3)	0·226 (3)	0·178 (2)
C(11)	0·5099 (3)	−0·0195 (3)	0·1455 (2)	H(10)	0·504 (3)	0·188 (3)	0·126 (2)
C(12)	0·4482 (3)	−0·1217 (3)	0·1737 (2)	H(11)	0·586 (3)	−0·040 (3)	0·122 (2)
C(13)	0·3348 (3)	−0·0983 (3)	0·2066 (1)	H(12)	0·487 (4)	−0·220 (3)	0·174 (2)
C(14)	0·1682 (2)	0·0576 (2)	0·3180 (1)	H(13)	0·291 (3)	−0·173 (3)	0·228 (2)
C(15)	0·3246 (2)	0·0646 (2)	0·3780 (1)	H(16)	0·444 (3)	0·158 (2)	0·483 (1)
C(16)	0·3403 (3)	0·1435 (3)	0·4486 (1)	H(17)	0·231 (3)	0·230 (3)	0·518 (2)
C(17)	0·2203 (3)	0·1756 (3)	0·4690 (2)	H(18)	−0·025 (3)	0·190 (3)	0·504 (1)
C(18)	0·0489 (3)	0·1498 (3)	0·4548 (2)	H(19)	−0·270 (3)	0·101 (2)	0·439 (1)
C(19)	−0·1902 (3)	0·0997 (3)	0·4157 (2)	H(20)	−0·305 (3)	−0·004 (2)	0·321 (1)
C(20)	−0·2163 (3)	0·0329 (3)	0·3451 (2)	H(21)	−0·124 (2)	−0·038 (2)	0·263 (1)

Table 2. *Bond lengths* (Å) *and angles* (°) *in* (I)

C(1)–C(2)	1·486 (3)	C(9)–C(10)	1·385 (4)	C(17)–C(23)	1·445 (4)
C(2)–C(3)	1·390 (3)	C(10)–C(11)	1·368 (5)	C(23)–C(18)	1·395 (4)
C(3)–C(4)	1·378 (4)	C(11)–C(12)	1·365 (4)	C(18)–C(19)	1·373 (4)
C(4)–C(5)	1·369 (4)	C(12)–C(13)	1·392 (4)	C(19)–C(20)	1·379 (4)
C(5)–C(6)	1·365 (4)	C(13)–C(8)	1·373 (4)	C(20)–C(21)	1·377 (4)
C(6)–C(7)	1·382 (4)	C(1)–C(14)	1·359 (3)	C(21)–C(22)	1·401 (3)
C(7)–C(2)	1·389 (3)	C(14)–C(15)	1·502 (3)	C(22)–C(23)	1·407 (3)
C(1)–C(8)	1·500 (3)	C(15)–C(16)	1·458 (4)	C(22)–C(14)	1·483 (3)
C(8)–C(9)	1·393 (4)	C(16)–C(17)	1·330 (4)	C(15)–O	1·221 (3)

C(2)–C(1)–C(8)	112·3 (2)	C(9)–C(10)–C(11)	120·6 (3)	C(22)–C(14)–C(15)	115·9 (2)
C(2)–C(1)–C(14)	124·4 (2)	C(10)–C(11)–C(12)	120·1 (3)	C(14)–C(15)–O	123·6 (2)
C(8)–C(1)–C(14)	123·2 (2)	C(11)–C(12)–C(13)	120·0 (3)	C(16)–C(15)–O	120·4 (2)
C(1)–C(2)–C(3)	120·5 (2)	C(12)–C(13)–C(8)	120·5 (2)	C(22)–C(23)–C(18)	120·1 (2)
C(2)–C(3)–C(4)	120·8 (2)	C(13)–C(8)–C(9)	119·1 (2)	C(23)–C(18)–C(19)	120·6 (3)
C(3)–C(4)–C(5)	120·3 (3)	C(1)–C(8)–C(13)	121·7 (2)	C(18)–C(19)–C(20)	119·8 (3)
C(4)–C(5)–C(6)	119·7 (3)	C(1)–C(14)–C(15)	118·8 (2)	C(19)–C(20)–C(21)	120·6 (3)
C(5)–C(6)–C(7)	120·7 (2)	C(14)–C(15)–C(16)	115·9 (2)	C(20)–C(21)–C(22)	121·0 (2)
C(6)–C(7)–C(2)	120·4 (2)	C(15)–C(16)–C(17)	121·2 (3)	C(21)–C(22)–C(23)	117·8 (2)
C(7)–C(2)–C(3)	118·0 (2)	C(16)–C(17)–C(23)	122·6 (3)	C(1)–C(14)–C(22)	124·9 (2)
C(1)–C(2)–C(7)	121·4 (2)	C(17)–C(23)–C(22)	119·6 (2)	C(21)–C(22)–C(14)	123·2 (2)
C(1)–C(8)–C(9)	119·1 (2)	C(23)–C(22)–C(14)	118·9 (2)	C(18)–C(23)–C(17)	120·2 (2)
C(8)–C(9)–C(10)	119·7 (2)				

Discussion. Bond lengths and angles of (I) are listed in Table 2 and a stereoscopic drawing of the molecule is given in Fig. 1. The bond lengths indicate that the molecule is well represented by structure (I). Torsion angles about the C(1)–C(14) bond are shown in Fig. 2. The most striking feature of this structure is the non-planarity of the naphthoquinone ring. In general, 1,2-naphthoquinones are planar (Bernstein, Cohen & Leiserowitz, 1974). However, there is considerable overcrowding in the C(2), C(8), C(1), C(14), C(15), O region of the molecule. This overcrowding manifests itself in a twist about the C(1)–C(14) exocyclic double bond (Fig. 2). In addition to this twist (torsion angles of 10·4 and 13·5°), there is an out-of-plane distortion at both C(1) and C(14); C(1) lies 0·031 Å out of the plane defined by C(2), C(8), and C(14), while C(14) lies 0·054 Å out of the plane defined by C(1), C(15), and C(22). The bond angles at C(1), C(14), and C(15) are all indicative of steric overcrowding. Weighted best-plane calculations (Table 3) through the phenyl rings,
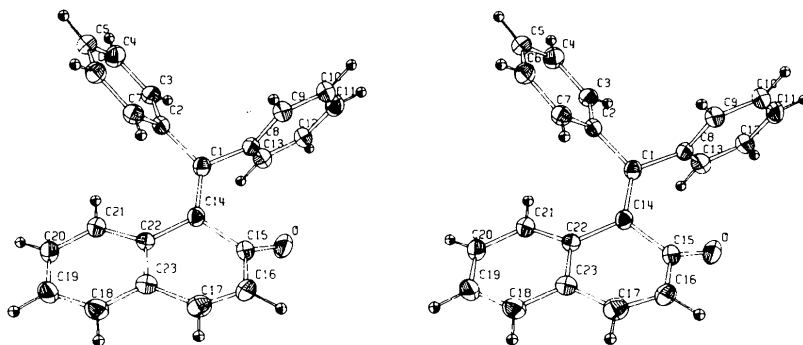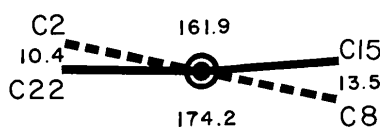
Fig. 1. Stereoscopic view of a single molecule of (I).

Fig. 2. Torsion angles (°) around the C(1)—C(14) bond in (I).

Table 3. *Some distances* (Å) *of atoms from various best planes in the molecule of* (I)

Atoms in the best-plane calculation were weighted as $1/\sigma^2$, where σ is the standard deviation from the least-squares results. Distances involving atoms included in the best-plane calculations are given in bold face.

	Distance	Distance		Distance	Distance
O	−0·493 (2)		C(2)	−0·010 (2)	
C(14)	0·140 (2)		C(3)	0·010 (3)	
C(15)	−0·150 (3)		C(4)	0·002 (3)	
C(16)	0·038 (3)		C(5)	−0·009 (3)	
C(17)	0·092 (3)		C(6)	0·003 (3)	
C(22)	−0·064 (2)	0·019 (2)	C(7)	0·006 (3)	
C(23)	−0·039 (2)	−0·019 (2)	C(8)		−0·008 (2)
C(18)		0·005 (3)	C(9)		0·016 (2)
C(19)		0·014 (3)	C(10)		−0·010 (3)
C(20)		−0·008 (3)	C(11)		−0·006 (3)
C(21)		−0·010 (2)	C(12)		0·011 (3)
			C(13)		−0·001 (3)
χ^2	10512	206		52	92
P^*	≪0·005	≪0·005		<0·005	<0·005

* The probability, based on the χ^2 test, that the points form a normal distribution.

C(2)—C(7) and C(8)—C(13), indicate significant nonplanarity which is greatly reduced when the atoms C(2) and C(8) respectively are removed from the calculations of the two planes.

In comparison with the corresponding bonds in a number of 1-diphenylmethylene-1,4-benzoquinones that have been studied in our laboratory (Lewis, 1977), the C(1)—C(14) [1·359 (3) Å] and C(15)—O [1·221 (3) Å] bond lengths reveal more double-bond character, while the C(14)—C(15) bond [1·502 (3) Å] exhibits more single-bond character; in the 1,4-benzoquinone derivatives the corresponding bond lengths are (average values for two structures) 1·379 (3), 1·243 (3), and 1·453 (3) Å. The ring bond between the two carbonyl groups in 1,2-naphthoquinones is typically quite long, 1·471–1·568 Å (Bernstein, Cohen & Leiserowitz, 1974). A view of the crystal packing is shown in Fig. 3. The closest intermolecular contact between non-hydrogen atoms is greater than 3·4 Å, indicating no strong intermolecular forces in the lattice.
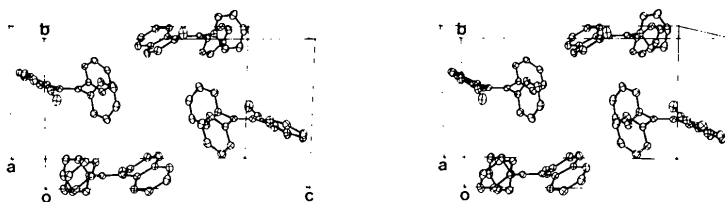
Fig. 3. Stereoscopic view of the packing of (I). The bonds in the reference molecule are darker.

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3-Phenyl-2-phenylimino-1,3-thiazetidine

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Abstract. $C_{14}H_{12}N_2S$, FW 240·3, monoclinic, space group $P2_1/c$, $a = 13·688$ (5), $b = 7·128$ (3), $c = 13·093$ (5) Å, $\beta = 108·09$ (5)°; $V = 1214·3$ Å^3, $Z = 4$, $D_c = 1·312$, $D_m = 1·305$ g cm^{-3}, Cu $K\alpha$ radiation, Ni filtered, μ(Cu $K\alpha$) = 6·1 cm^{-1}. Intensities were collected with the $\theta/2\theta$ scan mode and a scintillation counter. The structure was solved by symbolic addition methods from single-crystal diffractometer data to a final $R(F)$ of 0·031. The heterocycle of 1,3-thiazetidine is almost planar with an extremely small angle of 74·3° at S which introduces considerable strain into the ring. The three-coordinated N in the heterocycle has a tendency to adopt a pyramidal configuration which is reflected by a bend in the connexion between heterocycle and the adjacent benzene ring. Oxidation with H_2O_2/glacial acetic acid gives an enlargement of the ring to 1-oxo-1-λ^4-1,2,4-thiadiazolidin-3-one.

Introduction. The synthesis of 1,3-thiazetidine derivatives by various methods has been studied extensively over the last few years (see, for example, Mösinger, 1977). The title compound was obtained from a reaction of 1,3-diphenylthiourea with diiodomethane (Ried, Merkel & Mösinger, 1973). Oxidation of the thiazetidine ring results surprisingly in an enlargement to a five-membered ring (Ried, Mösinger

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& Schuckmann, 1976). The structure of the oxidation products is described in a second paper (Schuckmann, Fuess, Mösinger & Ried, 1978). The study of the structure of the thiazetidine system should cast some light on the nature of this oxidation reaction.

The compound was obtained in a 30% yield. Recrystallization from n-hexane gave colourless crystals.

Table 1. *Atom coordinates* $(\times 10^4)$, *with standard deviations in parentheses*

	x	y	z
C(1)	2965 (2)	2582 (3)	5270 (2)
C(2)	3394 (2)	3226 (4)	3838 (2)
C(3)	4870 (2)	2401 (3)	5528 (2)
C(4)	5202 (2)	2000 (4)	6622 (2)
C(5)	6214 (2)	1601 (4)	7129 (2)
C(6)	6919 (2)	1597 (4)	6575 (2)
C(7)	6599 (2)	2000 (4)	5503 (2)
C(8)	5585 (2)	2401 (3)	4975 (2)
C(9)	1841 (2)	1928 (4)	6198 (2)
C(10)	1209 (2)	3474 (5)	6093 (2)
C(11)	223 (3)	3235 (8)	6171 (2)
C(12)	−114 (3)	1517 (8)	6354 (2)
C(13)	510 (3)	−17 (8)	6462 (2)
C(14)	1485 (2)	199 (5)	6394 (2)
N(1)	3842 (1)	2806 (3)	4981 (1)
N(2)	2858 (1)	2084 (3)	6152 (1)
S	2107 (1)	3138 (1)	3976 (1)