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## Crystal Structures of 2,3,4 $\alpha$ $\beta$ ,6,7,8 $\alpha$ $\beta$ -Hexamethyl-4 $\alpha$ ,5,8,8 $\alpha$ -tetrahydro-1-naphthoquin-4 $\alpha$ (and 4 $\beta$ )-ol\*

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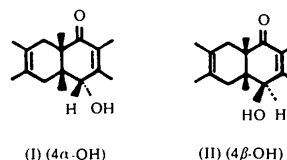
### Abstract

Crystals of the 4 $\alpha$ -ol are triclinic,  $P\bar{1}$ ,  $a = 7.660$  (1),  $b = 8.194$  (1),  $c = 12.981$  (2) Å,  $\alpha = 75.17$  (1),  $\beta = 79.57$  (1),  $\gamma = 65.19$  (1)°,  $Z = 2$ ,  $R = 0.043$  for 2438 reflexions. The 4 $\beta$ -ol is monoclinic,  $P2_1/n$ ,  $a = 7.671$  (1),  $b = 16.877$  (2),  $c = 10.994$  (1) Å,  $\beta = 92.27$  (2)°,  $Z = 4$ ,  $R = 0.041$  for 1640 reflexions. The molecules have different, ring-flipped conformations, so that in each molecule the hydroxyl group is pseudo-equatorial to the cyclohexenone ring. The two conformations produce differing intramolecular non-bonded contacts, which account for the varying photochemical reactivities in the solid state, and suggest that the photorearrangements in solution proceed from different conformations from those observed in the crystal lattice. Both structures contain intermolecular O(4)–H...O(1) hydrogen bonds.

### Introduction

A study of the photochemistry of various substituted 4 $\alpha$  $\beta$ ,5,8,8 $\alpha$  $\beta$ -tetrahydro-1-naphthoquin-4-ol systems [e.g. (I), (II)] has revealed reactivity differences, with either no solid-state reaction or formation of different photoproducts in solution and in the solid state (Appel,

Greenhough, Scheffer, Trotter & Walsh, 1980). To establish the reasons for these different results and to provide structural data which might indicate the factors influencing the reaction pathways, a structural study of the starting substrates has been undertaken. The present paper describes the structures of 2,3,4 $\alpha$  $\beta$ ,6,7,8 $\alpha$  $\beta$ -hexamethyl-4 $\alpha$ ,5,8,8 $\alpha$ -tetrahydro-1-naphthoquin-4 $\alpha$ -ol (I) and -4 $\beta$ -ol (II).



### Experimental

Recrystallization from petroleum ether/ethanol gave well formed colourless chunks of the 4 $\alpha$ -ol and large colourless plates of the 4 $\beta$ -ol; fragments were cut, of dimensions *ca* 0.3 × 0.3 × 0.3 mm and 0.6 × 0.2 × 0.2 mm, respectively. Preliminary unit-cell data were obtained from precession photographs, and accurate unit-cell parameters (Table 1) were determined by a least-squares analysis of the setting angles of 25 reflexions automatically located and centred on an Enraf–Nonius CAD-4 diffractometer ( $\theta = 12$ – $21^\circ$  for the 4 $\alpha$ -ol, 8– $12^\circ$  for the 4 $\beta$ -ol; graphite-monochromatized Mo  $K\alpha$  radiation). Space groups were

\* IUPAC names: 4 $\alpha$ (and 4 $\beta$ )-hydroxy-2,3,4 $\alpha$  $\beta$ ,6,7,8 $\alpha$  $\beta$ -hexamethyl-4 $\alpha$ ,5,8,8 $\alpha$ -tetrahydro-1(4H)-naphthalenone.

Table 1. *Experimental data*

	4 $\alpha$ -ol	4 $\beta$ -ol
Formula	C <sub>16</sub> H <sub>24</sub> O <sub>2</sub>	C <sub>16</sub> H <sub>24</sub> O <sub>2</sub>
Crystal system	Triclinic	Monoclinic
Space group	<i>P</i> 1 ( <i>C</i> <sub>1</sub> )	<i>P</i> 2 <sub>1</sub> / <i>n</i> ( <i>C</i> <sub>2h</sub> <sup>s</sup> )
<i>a</i> (Å)	7.660 (1)	7.671 (1)
<i>b</i> (Å)	8.194 (1)	16.877 (2)
<i>c</i> (Å)	12.981 (2)	10.994 (1)
$\alpha$ (°)	75.17 (1)	—
$\beta$ (°)	79.57 (1)	92.27 (2)
$\gamma$ (°)	65.19 (1)	—
<i>Z</i>	2	4
<i>D</i> <sub>x</sub> (Mg m <sup>-3</sup> )	1.16	1.16
Radiation	Mo <i>K</i> $\alpha$	Mo <i>K</i> $\alpha$
Monochromator	Graphite	Graphite
Scan	$\omega$ - $\theta$	$\omega$ - $\theta$
$\Delta\omega$ (°)	1.4 + 0.35 tan $\theta$	0.7 + 0.35 tan $\theta$
Aperture height (mm)	4	4
Aperture width (mm)	1.5 + 1.0 tan $\theta$	1.5 + 1.0 tan $\theta$
Maximum scan speed (deg min <sup>-1</sup> )	10.06	10.06
Prescan acceptance limit	30 $\sigma$	30 $\sigma$
Maximum counting time (s)	90	75
Maximum $\theta$ (°)	27.5	25
Number of check reflexions	3	3
Variation of checks (%)	$\pm 0.5$	$\pm 0.7$
Total reflexions	3254	2484
Reflexions >3 $\sigma$ *	2438 (75%)	1640 (66%)
<i>R</i>	0.043	0.041
<i>R</i> <sub>w</sub>	0.063	0.059
Goodness of fit	1.23	1.38

\*  $\sigma^2(I) = S + B + (0.04S)^2$ , *S* = scan count, *B* = background count.

determined from systematically absent reflexions, intensity statistics, and structure analyses.

The intensities were measured on the CAD-4 diffractometer; the experimental conditions are summarized in Table 1.

All non-hydrogen atoms were located by direct methods with use of *MULTAN* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), with 496 and 493  $|E|$  values > 1.55 and 1.45 for the 4 $\alpha$ -ol and 4 $\beta$ -ol, respectively, derived from a *K*-curve method (Ladd, 1978). Following anisotropic full-matrix least-squares refinements of C and O, the H atoms were located from difference maps and were assigned isotropic thermal parameters. Scattering factors for C and O were taken from Cromer & Mann (1968) and those for H from Stewart, Davidson & Simpson (1965). The final refinements included all atomic parameters, and the function minimized was  $\sum w(F_o - F_c)^2$ ,  $w = 1/\sigma^2(F)$ ; for the 4 $\beta$ -ol, an isotropic extinction parameter was refined, final value  $g = 1.9(6) \times 10^4$  (Becker & Coppens, 1974, 1975). Final values of  $R = \sum |\Delta F| / \sum |F_o|$  and  $R_w = [\sum w(F_o - F_c)^2 / \sum wF_o^2]^{1/2}$  for the reflexions >3 $\sigma$  are given in Table 1. The mean and maximum

shift/error on the final least-squares cycles were 0.01 and 0.23, respectively, for the 4 $\alpha$ -ol, and 0.06 and 0.74 for the 4 $\beta$ -ol; weighting analyses confirmed the suitability of the chosen weights. Difference Fourier syntheses after the final cycles showed random fluctuations of up to  $\pm 0.25$  e Å<sup>-3</sup>. Final positional parameters are listed in Table 2.\*

## Discussion

The tetrahydro-1-naphthoquin-4-ol ring system can exist in two low-energy conformations (*A* and *B* in Fig. 1); both have fused six-membered rings with half-chair conformations, and are folded and twisted about the C(4a)–C(8a) bond. They can be interconverted by ring ‘flipping’ involving single-bond rotations *via* a higher-energy conformer (*C* in Fig. 1), which has eclipsed bridgehead substituents and parallel double bonds. The flipping converts a pseudo-equatorial substituent at C(4) in the cyclohexenone ring of one conformer into a pseudo-axial substituent of the other conformer. These twist conformations are similar to that found in various substituted tetrahydro-1,4-naphthoquinones (Phillips & Trotter, 1977).

The molecular structures of the present hexamethyl-4 $\alpha$ - and -4 $\beta$ -ol derivatives (Fig. 2) show that the two molecules have different ring-flipped conformations, *A* for the 4 $\alpha$ -ol and *B* for the 4 $\beta$ -ol [to illustrate the similarity of the fused-ring systems in the two conformations, the molecules shown in Fig. 2 are enantiomorphic at the bridgehead C(4a) and C(8a)

\* Lists of structure factors, thermal parameters, bond distances involving hydrogen atoms, torsion angles, and packing diagrams for both compounds have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35181 (59 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

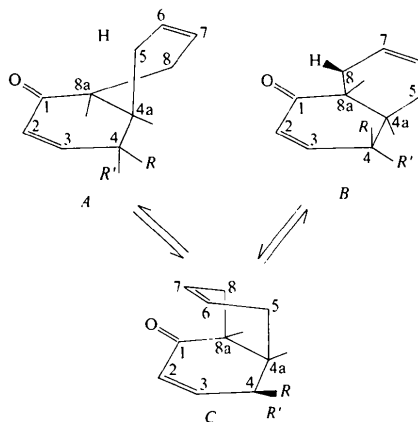


Fig. 1. Conformations of tetrahydro-1-naphthoquin-4-ols. Observed conformations in the crystal lattice are *A* for the 4 $\alpha$ -ol with *R* = OH, and *B* for the 4 $\beta$ -ol with *R*' = OH.

Table 2. Final positional parameters (fractional  $\times 10^5$ , for H  $\times 10^4$ ) with e.s.d.'s in parentheses

	4 $\alpha$ -ol (P $\bar{1}$ )			4 $\beta$ -ol (P2 $_1$ /n)		
	x	y	z	x	y	z
C(1)	84844 (16)	79109 (17)	34940 (10)	23525 (25)	29745 (13)	50503 (20)
C(2)	70973 (17)	75859 (17)	43917 (10)	30356 (26)	37019 (12)	56165 (19)
C(3)	54095 (17)	76387 (16)	42059 (10)	47694 (26)	38106 (12)	57773 (19)
C(4)	48850 (16)	78796 (17)	30899 (10)	60782 (25)	32220 (13)	53083 (22)
C(4a)	66129 (16)	75174 (16)	22438 (9)	53351 (24)	26658 (12)	43166 (18)
C(5)	78548 (17)	54446 (17)	24141 (11)	66313 (30)	19854 (14)	41331 (24)
C(6)	98250 (17)	49275 (18)	18236 (10)	67035 (26)	13571 (12)	51005 (22)
C(7)	106345 (18)	61379 (20)	14180 (11)	54877 (27)	12909 (12)	59240 (21)
C(8)	96365 (20)	81025 (20)	15396 (12)	39552 (28)	18451 (13)	59395 (22)
C(8a)	78643 (17)	85251 (17)	23569 (10)	36062 (23)	23105 (12)	47483 (19)
C(21)	76943 (27)	72481 (27)	54990 (12)	17010 (42)	42919 (19)	60185 (35)
C(31)	38719 (25)	74983 (26)	50877 (13)	55233 (43)	45070 (17)	64572 (31)
C(41)	58558 (24)	81759 (25)	11323 (12)	50404 (41)	31267 (20)	31214 (25)
C(61)	107939 (24)	29334 (23)	17496 (17)	82408 (40)	7984 (20)	50483 (43)
C(71)	125842 (27)	56841 (33)	7931 (19)	55028 (53)	6791 (21)	69200 (37)
C(81)	67616 (24)	106291 (20)	22021 (15)	27599 (37)	17529 (19)	37990 (31)
O(1)	100292 (14)	78187 (18)	36788 (9)	7758 (19)	28753 (11)	48730 (20)
O(4)	39954 (14)	66683 (15)	30720 (9)	75064 (21)	36710 (10)	48953 (22)
H(O4)	2806 (31)	7147 (27)	3220 (16)	8332 (44)	3380 (20)	4900 (28)
H(4)	3921 (20)	9199 (20)	2898 (12)	6444 (29)	2914 (13)	6000 (21)
H(51)	7157 (22)	4851 (22)	2168 (13)	6386 (31)	1744 (15)	3371 (24)
H(52)	7962 (21)	4950 (22)	3183 (13)	7834 (33)	2221 (14)	4077 (22)
H(81)	10548 (24)	8443 (22)	1726 (13)	2920 (32)	1561 (14)	6122 (20)
H(82)	9267 (26)	8909 (26)	872 (15)	4146 (30)	2216 (15)	6685 (23)
H(211)	9006 (40)	6842 (36)	5485 (22)	860 (46)	4036 (21)	6487 (32)
H(212)	7390 (40)	6226 (42)	6042 (25)	2192 (46)	4777 (25)	6208 (35)
H(213)	6947 (36)	8370 (37)	5813 (21)	977 (56)	4469 (26)	5387 (40)
H(311)	3984 (44)	6351 (47)	5401 (26)	4765 (58)	4760 (26)	6908 (43)
H(312)	3752 (46)	8134 (47)	5629 (27)	6499 (54)	4364 (23)	6972 (38)
H(313)	2551 (43)	8215 (40)	4884 (23)	6077 (55)	4861 (27)	5953 (43)
H(411)	6818 (28)	7927 (27)	585 (16)	4758 (38)	2757 (18)	2479 (29)
H(412)	5054 (28)	9550 (30)	983 (15)	6058 (42)	3422 (18)	2918 (26)
H(413)	5056 (34)	7607 (32)	1075 (18)	4138 (37)	3550 (17)	3204 (24)
H(611)	12129 (35)	2595 (32)	1497 (18)	8468 (50)	732 (23)	4219 (38)
H(612)	10154 (31)	2648 (29)	1290 (18)	7980 (50)	274 (26)	5362 (40)
H(613)	10530 (32)	2174 (33)	2425 (20)	9326 (57)	1038 (27)	5490 (38)
H(711)	12878 (37)	4741 (38)	425 (22)	6608 (56)	449 (26)	7039 (35)
H(712)	12572 (38)	6745 (40)	272 (24)	4746 (63)	283 (28)	6736 (45)
H(713)	13580 (38)	5285 (37)	1269 (22)	5160 (58)	909 (29)	7752 (45)
H(811)	5618 (24)	11043 (23)	2705 (14)	3521 (41)	1272 (21)	3600 (30)
H(812)	7697 (26)	11155 (26)	2279 (14)	1703 (43)	1540 (18)	4100 (26)
H(813)	6421 (27)	11087 (28)	1510 (18)	2392 (53)	2063 (23)	3064 (38)

atoms; the crystals are racemic]. The hydroxyl groups occupy the more sterically favoured pseudo-equatorial positions in both molecules, and this is probably the main feature which controls the conformations. The consequence of these conformations is that there is a close intramolecular non-bonded approach in the 4 $\alpha$ -ol of H(52) to the enone carbon atoms C(2) and C(3), with well separated, non-parallel C=C bonds; in the 4 $\beta$ -ol the close approach is from H(82) to C(2) and C(3). The degree of twist in the molecules is indicated by the bridgehead torsion angles: 4 $\alpha$ -ol, C(41)–C(4a)–C(8a)–C(81) = 59.9 (1), C(5)–C(4a)–C(8a)–C(1) = –63.2 (1)°; 4 $\beta$ -ol, C(41)–C(4a)–C(8a)–C(81) = 58.6 (2), C(4)–C(4a)–C(8a)–C(8) = –60.0 (2)°. The twists are thus similar in the two molecules, and to

those in the tetrahydronaphthoquinone analogue, 60.0 (6) and –61.4 (5)° (Phillips & Trotter, 1976).

In the 4 $\alpha$ -ol the C(2)···H(52) and C(3)···H(52) distances are 2.78 (2) and 2.72 (2) Å, respectively, not significantly different from each other, but shorter than the normal van der Waals contact distance of 2.90 Å. In the 4 $\beta$ -ol the C(2)···H(82) and C(3)···H(82) distances are 2.88 (2) and 2.92 (2) Å, respectively, significantly longer than the enone C···H(52) distances in the 4 $\alpha$ -ol, and comparable to the normal van der Waals distance. The solid-state photochemical reaction of the 4 $\alpha$ -ol proceeds *via* initial H(52) abstraction by the  $\beta$  enone carbon, C(3), while the 4 $\beta$ -ol is photochemically unreactive in the solid state (Appel, Greenhough, Scheffer, Trotter & Walsh, 1980).

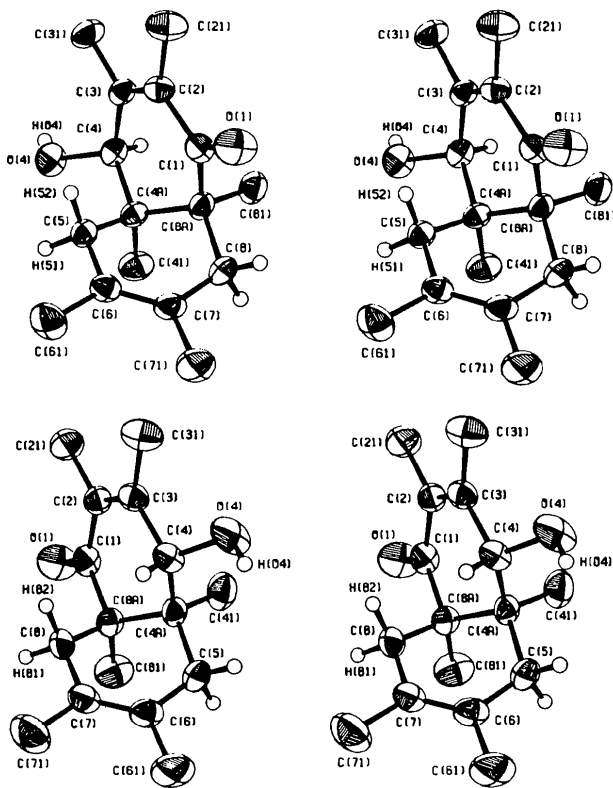


Fig. 2. Stereodiagrams of 2,3,4a $\beta$ ,6,7,8a $\beta$ -hexamethyl-4a,5,8,8a-tetrahydro-1-naphthoquin-4 $\alpha$ -ol (top) and -4 $\beta$ -ol (bottom). Thermal ellipsoids are at the 50% probability level, and methyl H atoms are omitted for clarity.

The  $C(2)-C(3)\cdots H(n)$  angle ( $\Delta_c$ ) and  $\tau_c$  [angle between the  $C(3)\cdots H(n)$  vector and the enone plane defined by  $Me-C(2)-C(3)-Me$ ] [ $H(n) = H(52)$  in the 4 $\alpha$ -ol, and  $H(82)$  in the 4 $\beta$ -ol] are quite similar in the two molecules, 78.5 and 53.2° in the 4 $\alpha$ -ol, and 75.2 and 49.3° in the 4 $\beta$ -ol. In the solid state the molecules have well separated, non-parallel  $C=C$  bonds;  $C(2)\cdots C(7)$  and  $C(3)\cdots C(6)$  intramolecular distances are 4.417 (2) and 4.400 (2) Å, respectively, for the 4 $\alpha$ -ol, and 4.490 (3) and 4.471 (3) Å for the 4 $\beta$ -ol, outside the limit of 4.1 Å suggested for successful 2 + 2 photocycloadditions (Schmidt, 1971; Scheffer & Dzakpasu, 1978). The  $O(1)\cdots H(81)$  intramolecular distance in the 4 $\alpha$ -ol is 2.45 (2) Å, similar to those observed in several tetrahydronaphthoquinone derivatives (Phillips & Trotter, 1977), where photochemical conversion is initiated by H abstraction by carbonyl oxygen (Scheffer & Dzakpasu, 1978); the 4 $\beta$ -ol conformer has no  $C=O\cdots H$  interaction geometrically favourable for H abstraction by oxygen, and in fact none of the tetrahydronaphthoquinols studied exhibit this pathway in solution or in the solid state (Appel, Greenhough, Scheffer, Trotter & Walsh, 1980).

The reactivity differences therefore appear to be a consequence of the short  $\beta-C\cdots H$  contacts in the 4 $\alpha$ -ol *vs* the longer distances in the 4 $\beta$ -ol, since the latter has no other primary photochemical reaction pathway available.

The photochemical reactions in solution proceed *via* intramolecular 2 + 2 cycloaddition for both molecules, probably due to reaction in solution *via* the higher-energy conformers *C*, which better fulfil the geometric

Table 3. Bond distances (Å) and angles (°)

	4 $\alpha$ -ol ( $\sigma =$ 0.002 Å)	4 $\beta$ -ol ( $\sigma =$ 0.003 Å)		4 $\alpha$ -ol ( $\sigma =$ 0.1°)	4 $\beta$ -ol ( $\sigma =$ 0.2°)		4 $\alpha$ -ol ( $\sigma =$ 0.1°)	4 $\beta$ -ol ( $\sigma =$ 0.2°)
$C(1)-C(2)$	1.479	1.464	$C(8a)-C(1)-C(2)$	118.8	119.4	$C(7)-C(6)-C(61)$	123.7	122.8
$C(2)-C(3)$	1.339	1.347	$C(8a)-C(1)-O(1)$	121.3	119.4	$C(6)-C(7)-C(8)$	121.8	121.7
$C(3)-C(4)$	1.516	1.517	$C(2)-C(1)-O(1)$	119.7	121.0	$C(6)-C(7)-C(71)$	123.8	124.6
$C(4)-C(4a)$	1.535	1.532	$C(1)-C(2)-C(3)$	120.6	120.3	$C(8)-C(7)-C(71)$	114.4	113.7
$C(4a)-C(5)$	1.538	1.537	$C(1)-C(2)-C(21)$	115.9	116.3	$C(7)-C(8)-C(8a)$	115.7	114.7
$C(5)-C(6)$	1.502	1.501	$C(3)-C(2)-C(21)$	123.5	123.4	$C(8)-C(8a)-C(1)$	110.6	106.2
$C(6)-C(7)$	1.333	1.330	$C(2)-C(3)-C(4)$	122.5	122.0	$C(8)-C(8a)-C(4a)$	109.8	109.6
$C(7)-C(8)$	1.502	1.503	$C(2)-C(3)-C(31)$	122.6	122.2	$C(4a)-C(8a)-C(81)$	113.4	112.0
$C(8)-C(8a)$	1.536	1.542	$C(4)-C(3)-C(31)$	114.9	115.9	$C(81)-C(8a)-C(8)$	108.7	108.7
$C(1)-C(8a)$	1.527	1.522	$C(3)-C(4)-C(4a)$	114.0	114.2	$C(81)-C(8a)-C(1)$	105.0	110.3
$C(4a)-C(8a)$	1.549	1.547	$C(3)-C(4)-O(4)$	110.9	106.8	$C(4a)-C(8a)-C(1)$	109.3	109.8
$C(1)-O(1)$	1.217	1.229	$C(4a)-C(4)-O(4)$	106.9	111.7			
$C(2)-C(21)$	1.510	1.507	$C(4)-C(4a)-C(5)$	109.4	109.1			
$C(3)-C(31)$	1.506	1.497	$C(4)-C(4a)-C(41)$	108.7	109.7			
$C(4)-O(4)$	1.427	1.421	$C(4)-C(4a)-C(8a)$	109.7	108.6			
$C(4a)-C(41)$	1.533	1.536	$C(41)-C(4a)-C(5)$	109.1	109.9			
$C(6)-C(61)$	1.506	1.513	$C(41)-C(4a)-C(8a)$	111.1	111.1			
$C(7)-C(71)$	1.509	1.505	$C(5)-C(4a)-C(8a)$	108.7	108.5			
$C(8a)-C(81)$	1.545	1.531	$C(4a)-C(5)-C(6)$	115.1	116.1			
			$C(5)-C(6)-C(7)$	122.3	122.3			
			$C(5)-C(6)-C(61)$	114.0	114.9			

requirements for intramolecular 2 + 2 cycloaddition than do the observed solid-state conformers *A* and *B*. The solid-state/solution reactivity differences in both the 4 $\alpha$ -ol and the 4 $\beta$ -ol thus appear to be due to crystal-lattice control of the photorearrangement *via* control of molecular conformation, as distinct from control in the sense of a least-motion process as found for 2,3,4 $\alpha\beta$ ,6,7 $\beta$ ,8 $\alpha\beta$ -hexamethyl-4a,7,8,8a-tetrahydro-1,4-naphthoquinone (Appel, Greenhough, Scheffer & Trotter, 1979; Greenhough & Trotter, 1979, 1980), where the completely different unimolecular photorearrangements in solution and the solid state are assumed to proceed from the same molecular conformation.

The bond lengths and angles in both molecules (Table 3) have normal values. The six-membered rings all have half-chair conformations: in the 4 $\alpha$ -ol molecule, C(4a) and C(8a) deviate by  $-0.48$  and  $0.25$  Å from the mean plane of C(1) to C(4), and by  $0.42$  and  $-0.33$  Å from the mean plane of C(5) to C(8); the corresponding displacements in the 4 $\beta$ -ol are  $-0.53$  and  $0.20$ , and  $0.33$  and  $-0.42$  Å. The carbonyl groups are only slightly non-planar, with C(1) out of plane by  $0.03$  Å ( $23\sigma$ ) in the 4 $\alpha$ -ol, and by  $0.02$  Å ( $9\sigma$ ) in the 4 $\beta$ -ol. The twist conformation produces two close intramolecular H $\cdots$ H contacts in the 4 $\beta$ -ol, H(52) $\cdots$ H(O4) =  $2.18$  (4) and H(82) $\cdots$ H(4) =  $2.27$  (3) Å; such short contacts are not present in the 4 $\alpha$ -ol.

Molecules in both crystals are linked into chains along *a* by O(4)–H(O4) $\cdots$ O(1) intermolecular hydrogen bonds, O(4) $\cdots$ O(1) =  $2.802$  (1) and  $2.846$  (2) Å in the 4 $\alpha$ -ol and 4 $\beta$ -ol, respectively, H(O4) $\cdots$ O(1) =  $1.98$  (2) and  $2.06$  (3) Å, O(4)–H(O4) $\cdots$ O(1) =  $171$  and  $167^\circ$ . Packing diagrams have been deposited.

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## Crystal Structures of 2,3,4 $\alpha\beta$ ,8 $\alpha\beta$ -Tetramethyl-4a,5,8,8a-tetrahydro-1-naphthoquin-4 $\beta$ -ol\* and Its Solid-State Photoproduct

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### Abstract

Crystals of the naphthoquinol are monoclinic,  $P2_1/n$ ,  $a = 7.319$  (2),  $b = 11.140$  (1),  $c = 15.510$  (3) Å,

\* IUPAC name: 4 $\beta$ -hydroxy-2,3,4 $\alpha\beta$ ,8 $\alpha\beta$ -tetramethyl-4a,5,8,8a-tetrahydro-1(4H)-naphthalenone.

$\beta = 96.41$  (3) $^\circ$ ,  $Z = 4$ ,  $R = 0.048$  for 931 reflexions. The photoproduct is triclinic,  $P\bar{1}$ ,  $a = 6.981$  (1),  $b = 7.619$  (2),  $c = 11.776$  (2) Å,  $\alpha = 77.70$  (1),  $\beta = 85.58$  (1),  $\gamma = 79.54$  (2) $^\circ$ ,  $Z = 2$ ,  $R = 0.070$  for 1126 reflexions. The naphthoquinol molecule has a conformation which has the hydroxyl group pseudo-