

bond angles 0.3°, and for torsion angles 0.5°. The C—H bond distances range from 0.833 to 1.33 Å and average 1.05 Å.

The methyl substituent is observed to be in the 7 α -position. For Fig. 3 a least-squares process (*FITMOL*, written by D. C. Rohrer for the *PROPHET* system) was used to optimize the overlap of the title compound and 17 β -hydroxy-4,14-estradien-3-one (Rohrer, Duax & Segaloff, 1978). The conformations of the two molecules are nearly identical. The principal difference is at C(7) where the interaction between the 7 β -methyl substituent and C(15) results in a puckering of the B ring. Hydrogen bonding from O(17) to O(3) [O(17)···O(3) = 2.81, O(17)—H = 0.97, O(3)···H = 1.922 Å, \langle O(17)—H—O(3) \rangle = 152°] links the molecules in chains extending parallel to the *ac* diagonal.

The crystal packing of the title compound is similar to that of 17 β -hydroxy-4,14-estradien-3-one. The cell dimensions of that compound are a = 9.7752, b = 25.519, c = 6.1158 Å and the hydrogen-bonded chains are of nearly identical orientations in the two crystals. The staggering of adjacent chains is adjusted and the

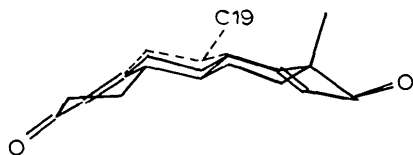


Fig. 3. A *PROPHET/FITMOL* overlap of 17 β -hydroxy-7 β -methyl-4,14-estradien-3-one (dashed line) and 17 β -hydroxy-4,14-estradien-3-one (solid line). The average interatomic separation is 0.05 Å. The maximum deviations are between the corresponding C(7), C(16) and C(15) atoms and have magnitudes of 0.17, 0.11 and 0.09 Å, respectively.

length of the *b* axis is expanded in order to accommodate the 7 β -methyl substituent which is oriented nearly parallel to the *b* axis. Complete stereo packing diagrams will appear in *Atlas of Steroid Structure*, Vol. II (Duax, Griffin & Weeks, 1980).

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Benzfurazan 1-Oxide

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Abstract. C₆H₄N₂O₂, triclinic, $C\bar{1}$, a = 14.073 (7), b = 6.772 (3), c = 7.515 (4) Å, α = 67.33 (3), β = 111.07 (3), γ = 90.93 (3)°, Z = 4, molecular volume = 0567-7408/79/123076-03\$01.00

152.6 Å³. (The standard setting has a = 7.515, b = 7.759, c = 6.772 Å, α = 114.94, β = 112.67, γ = 99.08°. The centered setting can be obtained from the © 1979 International Union of Crystallography

standard setting by the conversion $0\bar{2}1/001/\bar{1}00$.) The molecular dimensions are normal. The molecules are packed in layers with all intermolecular contacts within the layers being either $O\cdots H$ or $N\cdots H$.

Introduction. The structures of several substituted benzfuran 1-oxides have been determined previously (Britton & Noland, 1972; Gehrz & Britton, 1972, and references therein). We report here the structure of the unsubstituted compound. There are two preliminary reports on the crystal structure of this compound. Gol'der, Todres-Selektor & Bogdanov (1961) report a monoclinic unit cell in crystals obtained from aqueous solutions, and Hulme (1962) reports a triclinic cell in crystals obtained from ethanol solution. Our sample was supplied by Professor W. E. Noland; crystals suitable for the X-ray diffraction studies were prepared by sublimation. Our crystals were the same as those obtained by Hulme in all respects. The crystal used for data collection was coated with epoxy resin to prevent sublimation.

951 independent reflections were collected for $0 < \theta \leq 24^\circ$ on a Hilger & Watts four-circle automated diffractometer using Zr-filtered Mo $K\alpha$ radiation ($\lambda = 0.7107 \text{ \AA}$). All reflections were used in the subsequent calculations; $I = 0$ was used for those reflections with apparent negative intensities. The intensities of three check reflections did not change in any systematic way during the course of data collection. A trial structure was found from Patterson and Fourier maps and refined by full-matrix least squares with anisotropic thermal parameters for the heavy atoms and isotropic thermal parameters for the light atoms. The details of the experimental arrangement, the weighting scheme, and the programs used have all been described previously (Chow & Britton, 1974). Refinement converged with $R = 0.097$. The final R is large since all reflections were included in the refinement. A difference map calculated after refinement was complete had 0.20 e \AA^{-3} as the highest peak and 0.35 e \AA^{-3} as the lowest

trough. The final positional parameters are given in Table 1.*

Discussion. The bond distances and angles are given in Fig. 1. A rigid-body analysis was made using the method of Schomaker & Trueblood (1968); the r.m.s. difference between the observed U_{ij} values and those calculated from the rigid-body model is 0.0032 \AA^2 , about 50% larger than the average e.s.d. of the U_{ij} . The corrected and uncorrected bond distances are given in Table 2. The bond lengths and angles are similar to those found in 5-methylbenzfuran 1-oxide (Britton & Noland, 1972), and the discussion of the bonding in that compound is applicable here.

The packing is shown in Fig. 2. One of the reasons for determining this structure was our surprise in finding that the 5-methyl compound packed in layers.

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34670 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

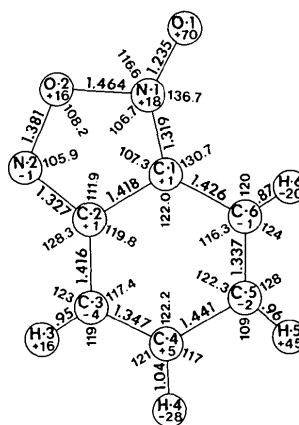


Fig. 1. Bond distances (\AA) and angles ($^\circ$) including corrections for rigid-body motions. The e.s.d.'s in the angles are 0.3° . The numbers in the circles are the distances (in 0.001 \AA) from the best plane through the benzene ring.

Table 1. Atomic coordinates ($\times 10^4$) and estimated standard deviations

	x	y	z
C(1)	-857 (2)	377 (5)	2378 (4)
C(2)	-378 (2)	-1601 (5)	2684 (4)
C(3)	645 (2)	-1785 (5)	2825 (5)
C(4)	1123 (3)	-42 (6)	2681 (5)
C(5)	625 (3)	1965 (5)	2359 (5)
C(6)	-341 (3)	2212 (5)	2210 (5)
N(1)	-1791 (2)	102 (4)	2322 (4)
N(2)	-998 (2)	-3087 (5)	2788 (5)
O(1)	-2531 (2)	1190 (4)	2169 (4)
O(2)	-1904 (2)	-2118 (4)	2582 (4)
H(3)	1007 (23)	-3090 (55)	3059 (48)
H(4)	1871 (24)	-74 (47)	2726 (45)
H(5)	1104 (29)	2993 (68)	2350 (58)
H(6)	-660 (22)	3413 (53)	2003 (45)

Table 2. Bond lengths (\AA)

	Uncorrected	Corrected for rigid-body motion
C(1)—C(2)	1.408 (4)	1.418
C(2)—C(3)	1.409 (5)	1.416
C(3)—C(4)	1.336 (5)	1.347
C(4)—C(5)	1.433 (6)	1.441
C(5)—C(6)	1.330 (5)	1.337
C(6)—C(1)	1.416 (4)	1.426
C(1)—N(1)	1.312 (4)	1.319
C(2)—N(2)	1.319 (4)	1.327
N(1)—O(1)	1.231 (5)	1.235
N(1)—O(2)	1.454 (4)	1.464
N(2)—O(2)	1.374 (4)	1.381

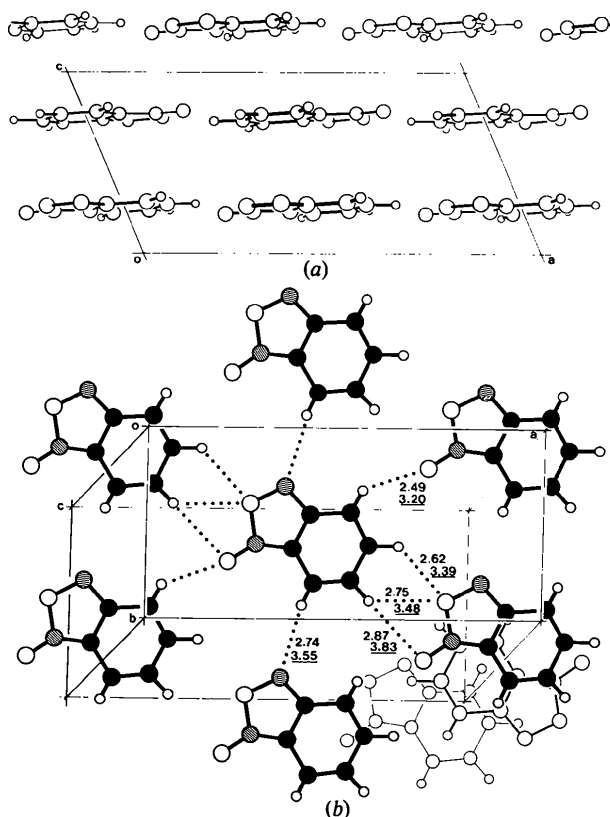


Fig. 2. The molecular packing. (a) Projection down b . (b) Projection perpendicular to the (001) plane. Short intermolecular contacts (\AA) within the layers of molecules are given. The upper number in each case is the $\text{O}\cdots\text{H}$ or $\text{N}\cdots\text{H}$ distance. The lower (underlined) number is the corresponding $\text{O}\cdots\text{C}$ or $\text{N}\cdots\text{C}$ distance. Molecules in the next two layers are shown in the lower right-hand corner of (b).

The same situation occurs here. The molecules occur in layers at $z \sim \frac{1}{4}$. The two sides of the layer make different contacts; the distance between layers is 3.37 \AA from $z \sim \frac{1}{4}$ to $z \sim \frac{3}{4}$, and 3.46 \AA and $z \sim \frac{1}{4}$ to $z \sim -\frac{1}{4}$. Individual molecules are tilted 10° with respect to the plane of the layers; this leads to corresponding perpendicular distances between the planes of the molecules of 3.39 and 3.47 \AA . The two kinds of contact between layers can be seen in the lower right-hand corner of Fig. 2. Within a layer all the close contacts are either $\text{O}\cdots\text{H}$ or $\text{N}\cdots\text{H}$. The distances, however, are not unusually short, so that we cannot describe these as $\text{O}\cdots\text{H}-\text{C}$ or $\text{N}\cdots\text{H}-\text{C}$ hydrogen bonds. Perhaps a satisfactory description would be to say they are incipient hydrogen bonds; the small polarity of the $\text{C}-\text{H}$ bond leads to enough interaction to determine the packing because the packing in layers is reasonably efficient in this case.

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3-Phenyl-1,3-thiazolidine-2,4-dione

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Abstract. $\text{C}_9\text{H}_7\text{NO}_2\text{S}$, monoclinic, space group $C2/c$, $a = 25.77$ (2), $b = 4.43$ (1), $c = 12.73$ (3) \AA , $\beta = 107.4$ (1) $^\circ$, $V = 1700 \text{ \AA}^3$, $Z = 8$, $M_r = 193.22$, $D_m = 1.49$ (floatation), $D_c = 1.51 \text{ Mg m}^{-3}$, $\mu(\text{Cu } K\alpha) = 2.89 \text{ mm}^{-1}$. The structure has been determined by direct

methods and refined by full-matrix least squares to an R of 0.056. The thiazolidine ring is slightly non-planar.

Introduction. The compound was obtained by Professor Pergal of the Institute of Chemistry, University