

map was calculated by the clockwise variation of two torsion angles $\theta_1 = \text{N2—C5—N2—C3}$ and $\theta_2 = \text{C9—N2—C5—N1}$, and energy minimization for each 15° rotation for θ_1 and θ_2 from -180 to 180°. The results of the calculation are illustrated in Fig. 2. All six conformational minima (marked on the map by the asterisks) are energetically equivalent; the differences not exceeding 1 kcal mol⁻¹ (1 kcal = 4.184 kJ). In spite of the absence of a global minimum, the conformation in the local minimum with $\theta_1 = -90$ and $\theta_2 = 65^\circ$ was accepted as most likely to exist in solution because the crystallographic conformation with the relative configuration of chiral center *R* (marked on the map by the black square) is close to the selected minimum and the energy barriers are equal to 9.2 and 14.6 kcal mol⁻¹ respectively for clockwise rotation of the θ_1 and θ_2 angles.

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Structure of 3-Hydroxyphenalenone

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Abstract. 3-Hydroxyphenalenone (enol form), C₁₃H₈O₂, *M_r* = 196.21, monoclinic, *Pc*, *a* = 9.089 (2), *b* = 17.737 (2), *c* = 14.265 (2) Å, β = 125.65 (2)°, *V* = 1868.5 (5) Å³, *Z* = 8, *D_x* = 1.40 g cm⁻³, $\lambda(\text{Mo K}\alpha)$ =

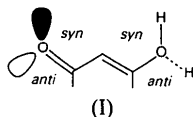
0.71069 Å, μ = 0.876 cm⁻¹, *F*(000) = 816, *T* = 290 K, *R* = 0.050 for 3182 independent reflections. The crystal structure is characterized by one-dimensional chains of hydrogen-bonded enol molecules. The intermolecular hydrogen bond in a *syn-syn* configuration has a short intermolecular oxygen distance (average 2.57 Å). All the enone

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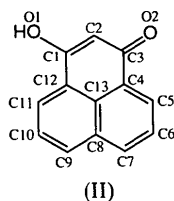
moieties arrange in almost the same direction along the chain: this leads to a polar crystal through the one-dimensional alignment of the dipole moments.

Introduction. The crystal structures of 3-hydroxyenones have drawn attention from the viewpoint of three-dimensional configurations connected with intermolecular hydrogen bonds. As summarized in Table 1, four kinds of configurations with respect to the relative orientation of the —O—H moiety and the =O...H moiety have been reported for 3-hydroxyenones (I).



A phenalenyl ring system with a O=C—C=C—OH moiety is interesting not only from the structural point of view but also because of the possibility of proton transfer along the hydrogen bond in the solid state (Svensson & Abrahams, 1986). The activation energy of the proton transfer in solid 3-hydroxyphenalenone could be small because both the protonated and deprotonated species are stabilized, based on the characteristics of the phenalenyl structure (Matsumiya, Izuoka & Sugawara, 1990). Thus intermolecular proton transfer coupled with tautomerization of O=C—C=C—OH moieties might be realized in the solid state.

In this paper we report the X-ray crystal structure of the title compound (II), focusing on the geometry of the hydrogen bonds and also on the possibility of proton transfer in the crystal.



Experimental. Compound (II) crystallized as a brownish yellow prism with dimensions 0.70 × 0.30 × 0.30 mm from acetic acid solution (m.p. 490 K). Monoclinic, space group *Pc*. Data collection: Rigaku AFC-5 four-circle diffractometer, graphite-monochromated Mo *K*α radiation; cell parameters refined with 25 strong reflections (20 < 2θ < 25°), ω–2θ scan, scan speed 4° min⁻¹ in ω. The intensities of three standard reflections (012, 360 and 481) showed no significant variations; 4713 reflections measured, 3188 independent reflections (4 < 2θ < 55°; -15 ≤ *h* ≤ 15, 0 ≤ *k* ≤ 23, 0 ≤ *l* ≤ 12) with |*F*_o| > 3σ(*F*_o) were used for the analysis; *R*_{int} = 0.032;

Table 1. Configurations and O—O distances of hydrogen bonds in several 3-hydroxyenones

Configuration	Compound	O—O (Å)	Reference
(1) <i>syn-syn</i> (planar) (helix)	3-Hydroxyphenalenone	2.57 (av.)	This work Semmingsen (1974) Singh & Calvo (1985)
	Dimedone	2.593 (2)	
(2) <i>syn-anti</i> (planar, cyclic)	6:1 Cyclohexanedione: benzene cyclamer	2.579 (1)	Etter <i>et al.</i> (1986),
(3) <i>anti-syn</i> (planar)	2-Methyl-1,3-cyclopentadione (MCPD)	2.598 (2)	Katrusiak (1989)
(4) <i>anti-anti</i> (planar) (planar) (planar)	1,3-Cyclohexanedione	2.561 (4)	Etter <i>et al.</i> (1986) Katrusiak (1990) Semmingsen (1977)
	1,3-Cyclopentanedione	2.542 (8)	
	Phenylmalonaldehyde	2.575 (2)	

data corrected for Lorentz and polarization factors, no absorption correction applied ($\mu r < 1$). Six very strong reflections excluded in the refinements. Since not all the H atoms were found from the ΔF map, refinements were performed placing vinylic and aromatic protons at calculated positions, the positions of hydroxyl protons were revealed in the final ΔF map; positional parameters and *B*_{iso} values (6.0 Å²) of H atoms were fixed. 540 variable parameters, quantity minimized $\sum w(|F_o| - |F_c|)^2$, *R* = 0.050, *wR* = 0.062, *w* = 0.5 (|*F*_o| < 3.5), 1.0 (3.5 < |*F*_o| < 12.0) and 144.0/|*F*_o|² (12.0 < |*F*_o|), maximum | $\Delta\rho$ | = 0.25 e Å⁻³, maximum (Δ/σ) = 0.11. The atomic coordinates and *B*_{eq} values for non-H atoms are listed in Table 2.* Structure solved by direct methods using *SAPI85* (Fan, Qian, Yao, Zheng & Hao, 1988), non-H atoms refined anisotropically by block-diagonal least squares; scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations carried out on a FACOM A-70 computer with *UNICSIII* (Sakurai & Kobayashi, 1979).

Although molecules *A* and *D* are placed in a manner such that they are nearly related by a two-fold screw axis, as are *B* and *C*, the observation of second harmonic generation eliminates the possibility of the centrosymmetric space group *P2₁/c*.

Discussion. The crystal structure determined for 3-hydroxyphenalenone is depicted in Fig. 1. There are four independent molecules (*A*, *B*, *C*, *D*), constituting two kinds of one-dimensional in-plane chains (*A*–*B*–*A*–*B*–, *C*–*D*–*C*–*D*–). The molecular axis through the C(2) and C(8) atoms for molecule *A* and that for *B* are almost parallel to each other, while the corresponding axes for *C* and *D* are tilted by 9.4°. The geometry of the hydrogen bonds is in a *syn-syn*

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54640 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0524]

Table 2. Fractional atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters (\AA^2) with *e.s.d.*'s in parentheses

$$B_{eq} = (4/3) \sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
Molecule A				
O(1)	5036	2567 (2)	6541	5.0 (1)
O(2)	-214 (4)	2757 (2)	6460 (3)	4.5 (1)
C(1)	3298 (4)	2437 (2)	6085 (3)	3.2 (1)
C(2)	2443 (5)	2700 (2)	6539 (3)	3.4 (1)
C(3)	590 (5)	2534 (2)	6028 (3)	3.3 (1)
C(4)	-435 (5)	2094 (2)	4946 (3)	3.0 (1)
C(5)	-2242 (5)	1949 (2)	4384 (3)	3.9 (2)
C(6)	-3189 (5)	1536 (3)	3349 (4)	5.0 (2)
C(7)	-2319 (6)	1261 (3)	2911 (4)	4.7 (2)
C(8)	-453 (6)	1387 (2)	3456 (3)	3.8 (2)
C(9)	496 (6)	1113 (2)	3031 (4)	4.6 (2)
C(10)	2300 (7)	1250 (3)	3587 (4)	4.7 (2)
C(11)	3242 (5)	1692 (2)	4584 (3)	3.9 (2)
C(12)	2382 (5)	1971 (2)	5045 (3)	3.1 (1)
C(13)	509 (5)	1818 (2)	4488 (3)	3.0 (1)
Molecule B				
O(1)	1759 (4)	3385 (2)	8454 (2)	4.2 (1)
O(2)	-3652 (4)	3553 (2)	8181 (3)	4.9 (1)
C(1)	767 (5)	3628 (2)	8801 (3)	3.3 (1)
C(2)	-997 (5)	3470 (2)	8257 (3)	3.5 (1)
C(3)	-2031 (5)	3736 (2)	8643 (3)	3.4 (1)
C(4)	-1113 (5)	4233 (2)	9673 (3)	3.4 (1)
C(5)	-2055 (6)	4536 (3)	10066 (4)	4.7 (2)
C(6)	-1163 (8)	5013 (3)	11044 (4)	6.1 (3)
C(7)	638 (7)	5165 (2)	11611 (4)	5.4 (2)
C(8)	1659 (6)	4866 (2)	11235 (3)	4.1 (2)
C(9)	3507 (7)	5018 (3)	11800 (4)	5.5 (2)
C(10)	4425 (6)	4718 (3)	11412 (4)	5.6 (2)
C(11)	3568 (5)	4255 (2)	10426 (4)	4.3 (2)
C(12)	1745 (5)	4098 (2)	9842 (3)	3.2 (1)
C(13)	737 (5)	4392 (2)	10238 (3)	3.2 (1)
Molecule C				
O(1)	5370 (3)	1384 (2)	8683 (2)	4.3 (1)
O(2)	346 (4)	1643 (2)	8820 (2)	4.1 (1)
C(1)	3679 (4)	1246 (2)	8308 (3)	3.1 (1)
C(2)	2906 (5)	1533 (2)	8820 (3)	3.1 (1)
C(3)	1055 (4)	1387 (2)	8351 (3)	2.8 (1)
C(4)	-32 (5)	938 (2)	7293 (3)	3.1 (1)
C(5)	-1845 (6)	824 (2)	6782 (4)	4.2 (2)
C(6)	-2888 (5)	371 (3)	5769 (4)	4.7 (2)
C(7)	-2067 (6)	49 (2)	5307 (3)	4.6 (2)
C(8)	-217 (6)	164 (2)	5799 (3)	4.1 (2)
C(9)	677 (7)	-173 (2)	5360 (4)	5.1 (2)
C(10)	2470 (7)	-56 (3)	5869 (4)	5.0 (2)
C(11)	3493 (6)	406 (2)	6840 (4)	4.3 (2)
C(12)	2677 (5)	738 (2)	7314 (3)	3.3 (1)
C(13)	820 (5)	615 (2)	6813 (3)	3.0 (1)
Molecule D				
O(1)	2298 (4)	2304 (2)	817 (2)	4.1 (1)
O(2)	-3263 (4)	2318 (2)	362 (3)	5.3 (1)
C(1)	1231 (5)	2491 (2)	1125 (3)	3.0 (1)
C(2)	-544 (5)	2299 (2)	519 (3)	3.3 (1)
C(3)	-1652 (5)	2520 (2)	888 (3)	3.8 (1)
C(4)	-830 (5)	2987 (2)	1924 (3)	3.4 (1)
C(5)	-1865 (6)	3241 (3)	2296 (4)	4.4 (2)
C(6)	-1034 (7)	3676 (3)	3307 (4)	5.0 (2)
C(7)	761 (7)	3857 (2)	3936 (4)	4.8 (2)
C(8)	1847 (6)	3614 (2)	3572 (3)	3.8 (2)
C(9)	3714 (7)	3781 (3)	4215 (4)	5.0 (2)
C(10)	4727 (6)	3545 (3)	3836 (4)	4.9 (2)
C(11)	3914 (5)	3123 (2)	2819 (3)	3.9 (2)
C(12)	2091 (5)	2944 (2)	2184 (3)	3.2 (1)
C(13)	1017 (5)	3178 (2)	2550 (3)	3.0 (1)

Table 3. Interatomic distances (\AA) and angles ($^\circ$) with *e.s.d.*'s in parentheses

	Mol. A	Mol. B	Mol. C	Mol. D
O(1)—C(1)	1.331 (5)	1.328 (6)	1.322 (5)	1.319 (5)
O(2)—C(3)	1.264 (6)	1.256 (6)	1.254 (5)	1.247 (6)
C(1)—C(2)	1.352 (6)	1.342 (7)	1.374 (6)	1.357 (6)
C(1)—C(12)	1.462 (6)	1.467 (6)	1.464 (6)	1.471 (6)
C(2)—C(3)	1.423 (7)	1.418 (6)	1.426 (6)	1.436 (6)
C(3)—C(4)	1.477 (6)	1.484 (6)	1.466 (6)	1.463 (7)
C(4)—C(5)	1.368 (6)	1.377 (7)	1.374 (7)	1.397 (7)
C(4)—C(13)	1.434 (6)	1.406 (7)	1.419 (6)	1.409 (6)
C(5)—C(6)	1.406 (7)	1.415 (9)	1.427 (7)	1.405 (8)
C(6)—C(7)	1.353 (8)	1.365 (10)	1.375 (8)	1.365 (9)
C(7)—C(8)	1.412 (8)	1.417 (9)	1.409 (8)	1.423 (8)
C(8)—C(9)	1.399 (8)	1.402 (8)	1.416 (9)	1.412 (8)
C(8)—C(13)	1.420 (7)	1.429 (7)	1.425 (7)	1.418 (7)
C(9)—C(10)	1.364 (8)	1.350 (9)	1.359 (9)	1.378 (8)
C(10)—C(11)	1.396 (8)	1.408 (8)	1.397 (8)	1.399 (7)
C(11)—C(12)	1.374 (7)	1.381 (7)	1.392 (7)	1.385 (7)
C(12)—C(13)	1.423 (6)	1.426 (6)	1.415 (6)	1.411 (6)
O(1)—C(1)—C(2)	123.8 (4)	124.0 (4)	123.4 (4)	123.9 (4)
O(1)—C(1)—C(12)	113.9 (4)	115.2 (4)	115.2 (4)	115.2 (4)
C(2)—C(1)—C(12)	122.3 (4)	121.3 (4)	121.4 (4)	121.0 (4)
C(1)—C(2)—C(3)	121.5 (4)	123.0 (4)	120.8 (4)	121.7 (4)
O(2)—C(3)—C(2)	122.3 (4)	123.4 (4)	121.1 (4)	122.0 (4)
O(2)—C(3)—C(4)	119.0 (4)	118.9 (4)	119.9 (4)	120.0 (4)
C(2)—C(3)—C(4)	118.8 (4)	117.7 (4)	119.0 (4)	118.1 (4)
C(3)—C(4)—C(5)	121.5 (4)	120.7 (4)	120.4 (4)	120.4 (4)
C(3)—C(4)—C(13)	118.4 (4)	118.4 (4)	119.1 (4)	119.7 (4)
C(5)—C(4)—C(13)	120.1 (4)	120.5 (5)	120.5 (4)	119.9 (4)
C(4)—C(5)—C(6)	120.3 (5)	120.0 (6)	120.2 (5)	119.3 (5)
C(5)—C(6)—C(7)	120.5 (5)	120.2 (6)	119.6 (5)	121.7 (6)
C(6)—C(7)—C(8)	121.8 (5)	121.7 (6)	121.6 (5)	120.4 (6)
C(7)—C(8)—C(9)	123.5 (5)	122.7 (5)	123.0 (5)	121.7 (5)
C(7)—C(8)—C(13)	118.2 (5)	117.6 (5)	118.6 (5)	118.3 (5)
C(9)—C(8)—C(13)	118.3 (5)	119.7 (5)	118.4 (5)	120.0 (5)
C(8)—C(9)—C(10)	121.5 (5)	120.6 (5)	121.2 (6)	120.4 (5)
C(9)—C(10)—C(11)	120.6 (5)	121.7 (5)	121.2 (6)	120.0 (5)
C(10)—C(11)—C(12)	120.5 (5)	119.4 (5)	121.6 (4)	121.0 (4)
C(1)—C(12)—C(11)	122.9 (4)	122.1 (4)	121.6 (4)	121.0 (4)
C(1)—C(12)—C(13)	117.6 (4)	117.3 (4)	118.0 (4)	118.2 (4)
C(11)—C(12)—C(13)	119.5 (4)	120.6 (4)	120.4 (4)	120.8 (4)
C(4)—C(13)—C(8)	119.0 (4)	120.0 (4)	119.5 (4)	120.4 (4)
C(4)—C(13)—C(12)	121.3 (4)	121.9 (4)	121.3 (4)	121.4 (4)
C(8)—C(13)—C(12)	119.7 (4)	118.0 (4)	119.2 (4)	118.3 (4)

Intermolecular O—O distances (\AA)

O(1A)···O(2B ⁱ)	2.589 (5)	O(1C)···O(2D ⁱⁱⁱ)	2.560 (5)
O(1B)···O(2A ⁱ)	2.568 (5)	O(1D)···O(2C ⁱⁱ)	2.597 (5)

Symmetry code: (i) $x+1, y, z$; (ii) x, y, z ; (iii) $x+1, y, z+1$; (iv) $x, y, z-1$.

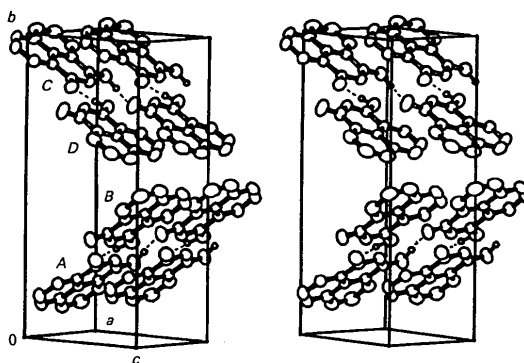


Fig. 1. Stereoview of the packing of 3-hydroxyphenalenone (ORTEP; Johnson, 1965). The thermal ellipsoids are drawn at the 50% probability level, excluding H atoms. Hydrogen bonds are represented as dashed lines.

configuration, which is similar to the case of dimedone (Table 1). The short intermolecular O—O distances, 2.568 (5) and 2.589 (5) \AA for the A—B chain, 2.560 (5) and 2.597 (5) \AA for the C—D chain, indicate the presence of strong hydrogen bonds. The O—O distances are even slightly shorter than that of

dimedone, 2.593 (2) \AA . Hydrogen bonds in 3-hydroxyphenalenones are generally classified as of the strong type (Emsley, 1980).

The bond lengths and the bond angles of the molecules are listed in Table 3. The average bond lengths in the O=C—C=C—OH moiety from O(2) to O(1) are 1.26, 1.43, 1.36 and 1.33 Å for *A*, *B*, *C* and *D*, respectively. They are similar to those observed in dimedone and MCPD. The structure of the naphthalene moiety does not differ significantly from that of naphthalene.

The dipole moments of the *A*–*B* and *C*–*D* chains, which originate in the O=C—C=C—OH moieties, are directed towards the same direction along the *a* axis, resulting in the polarity of the crystal. The situation is similar to the case of phenylmalonaldehyde (Semmingsen, 1977). Thus the crystal may show ferroelectric properties. However, a dielectric response of the crystal was not observed in the temperature range 300–450 K under 10 kHz modulation. Neither dynamic processes nor phase transitions were observed in the solid-state ¹³C NMR (CP/MAS) measurement even when the temperature was raised to 393 K. Therefore, it was concluded that the hydroxyl protons are tightly fixed at a particular site of the hydrogen bond even at high temperatures.

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Structure of 5-(1-Methyl-2,1-benzisothiazol-3-ylideneamino)-2,4-pentadienal

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Abstract. C₁₃H₁₂N₂OS, *M_r* = 244.31, monoclinic, *P*2₁/*a*, *a* = 7.743 (2), *b* = 12.430 (5), *c* = 12.776 (5) Å, β = 90.52 (3)°, *V* = 1229.6 (7) Å³, *Z* = 4, *D_x* = 1.32 Mg m⁻³, λ(Mo Kα) = 0.71069 Å, graphite monochromator, μ = 0.236 mm⁻¹, *F*(000) = 512, *T* = 295 K, *R* = 0.040, *wR* = 0.049 for 2958 independent reflections collected. This is only the third example of a crystal structure containing a 2,1-benzisothiazole ring system. Bond lengths in the isothiazole ring of the title compound suggest much less double-bond character than observed in the other two known structures. The molecule is essentially planar and packs in the crystal with the benzisothiazole rings stacked in the [100] direction.

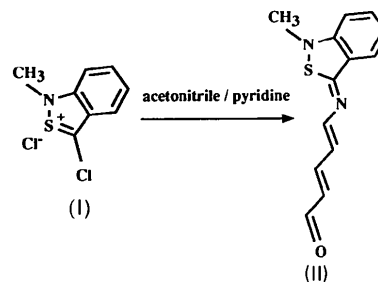
Introduction. As part of ongoing studies of benzisothiazoles, an attempt was made to form a 2,1-

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benzisothiazol-3-ylideneacetone nitrile by treating 3-chloro-1-methyl-2,1-benzisothiazolium chloride (I) with acetonitrile in pyridine. Instead, the title compound (II) was afforded on work up.



Experimental. 3-Chloro-1-methyl-2,1-benzisothiazolium chloride (0.220 g, 1.0 mmol) in acetonitrile (10 mL) and pyridine (2 mL) was boiled for 15 min.

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