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HYDROGEN-BONDED RIBBON STRUCTURE IN 2-PHENYL-3-HYDROXYPHENALENONE

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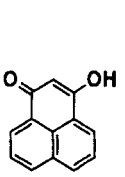
Abstract The title compound crystallized in the triclinic space group $P\bar{1}$. The crystal structure contains intermolecular hydrogen-bonded ribbon structure, though 2-position of the phenalenyl ring is substituted by a bulky phenyl group. The O···O distances of hydrogen-bonds are 2.734(3) and 2.718(3) Å. The phenyl ring rotates by 51.52 and 54.69° toward the phenalenyl ring.

INTRODUCTION

We have recently reported the solid state properties of the charge transfer complexes having hydrogen-bonded (H-bonded) chains.[1] Such H-bonded charge transfer (HBCT) systems composed of the two components of a proton-electron donor and a proton-electron acceptor are expected to show characteristic solid state properties such as cooperative proton-electron transfer phenomena. In order to expand examples of the molecular assemblies of HBCT systems, it is interesting to know the molecular packing of molecules which contain both the proton donating and accepting groups in a single molecular skeleton. The molecules having the hydroxyenone moiety, O=C–C=C–O–H, are suitable for such a single H-bonded system.

Recently, the crystal structure of 3-hydroxyphenalenone **1**, which is classified into one of the single H-bonded systems, has been reported.[2] Now, we undertook the X-ray crystal structure determination of 2-phenyl-3-hydroxyphenalenone **2** in order to gain insight into the effect of a bulky phenyl group at 2-position of phenalenone moiety on the molecular packing, especially on the intermolecular H-bonding pattern.



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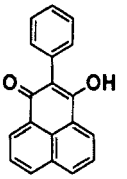

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RESULTS AND DISCUSSION

Experimental

The title compound was prepared by the literature procedure[3] and purified by the recrystallization from tetrahydrofurane to give orange plates with mp 226.5-227.5 °C. Crystal data were collected on an Enraf-Nonius CAD4 diffractometer with graphite monochromated Cu $K\alpha$ radiation (λ = 1.5418 Å). The structure was solved by the direct method of Xtal 3.0 program system[4] and refined on F^2 by a full-matrix least-squares technique. The crystal data and the structure determination parameters are listed in Table I.

TABLE I Crystal data and structure determination parameters of **2**.

Crystal data		Structure determination parameters	
molecular formura	$C_{19}H_{12}O_2 \cdot C_{19}H_{12}O_2$	μ (Cu$K\alpha$)	7.26 cm^{-1}
formura weight	544.61	scan type	ω–2θ
crystal system	triclinic	$2\theta_{max}$	139.8
space group	$P\bar{1}$ (No.2)	range of h,k,l measured	$\pm$15/$\pm$17/0,9
a, Å	12.553(3)	no. of unique reflections measured	4817 (R_{int} = 0.013)
b, Å	14.536(9)		
c, Å	7.569(1)	no of observations (I > 3.00$\sigma(I)$)	3308
α, deg	90.76(3)		
β, deg	99.06(2)	residuals: R;Rw	0.048 ; 0.047
γ, deg	109.44(4)	goodness of fit indicator	1.14
V, Å^3	1283.0(9)	final max/min $\Delta\rho$, e/Å^3	0.22/-0.24
Z value	2		
D(calcd)/Mg m^{-3}	1.410		

Molecular Structure

The molecular structure of **2** with the atomic numbering scheme is shown in Figure 1. Bond distances of the phenalenyl ring are given in Table II. The phenalenyl rings are nearly planar with maximum deviation of 0.043 (3) for C2 and 0.040 (3) Å for C21 from the least square plane. The phenyl ring rotates by 51.52 and 54.69° of a dihedral angle toward the phenalenyl ring because of the steric repulsion between the phenyl ring and the two oxygen atoms. The bond lengths of 3-hydroxyenone moiety, O=C–C=C–O–H, are almost similar to those of parent 3-hydroxyphenalenone.

Crystal Structure

The molecular packing is shown in Figure 2. The molecules are linked by an O–H···O type H-bond between the hydroxyl and the carbonyl groups with the O···O distance of 2.734 (3) and 2.718 (3) Å, in spite of the presence of the phenyl group. The H-bonds

TABLE II Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	Beq
O(1)	0.9851(2)	0.4874(1)	0.7277(3)	3.67(5)
O(2)	0.9600(2)	0.7994(1)	0.7657(3)	3.40(5)
O(3)	1.0196(2)	0.9972(1)	0.7698(3)	3.62(5)
O(4)	1.0463(2)	1.3236(1)	0.7326(3)	3.33(5)
C(1)	1.0297(2)	0.5770(2)	0.7190(4)	2.72(6)
C(2)	0.9661(2)	0.6426(2)	0.7366(4)	2.51(5)
C(3)	1.0201(2)	0.7417(2)	0.7353(4)	2.56(6)
C(4)	1.1379(2)	0.7849(2)	0.7029(4)	2.67(6)
C(5)	1.1885(3)	0.8841(2)	0.6913(4)	3.39(7)
C(6)	1.3017(3)	0.9221(2)	0.6537(5)	3.93(7)
C(7)	1.3616(3)	0.8620(2)	0.6305(5)	3.71(7)
C(8)	1.3135(2)	0.7602(2)	0.6445(4)	3.11(6)
C(9)	1.3742(3)	0.6964(2)	0.6232(5)	3.88(8)
C(10)	1.3241(3)	0.5978(3)	0.6283(5)	4.14(8)
C(11)	1.2108(3)	0.5586(2)	0.6598(5)	3.61(7)
C(12)	1.1494(2)	0.6191(2)	0.6861(4)	2.71(6)
C(13)	1.1994(2)	0.7209(2)	0.6783(4)	2.57(6)
C(14)	0.8428(2)	0.6012(2)	0.7539(4)	2.59(6)
C(15)	0.8056(3)	0.5352(2)	0.8818(4)	3.04(6)
C(16)	0.6899(3)	0.4984(2)	0.8954(4)	3.55(7)
C(17)	0.6103(3)	0.5263(2)	0.7827(5)	3.84(7)
C(18)	0.6458(3)	0.5914(2)	0.6536(5)	3.79(7)
C(19)	0.7614(2)	0.6287(2)	0.6397(4)	3.16(6)
C(20)	0.9758(2)	1.0617(2)	0.7817(4)	2.59(6)
C(21)	1.0390(2)	1.1636(2)	0.7626(4)	2.49(5)
C(22)	0.9856(2)	1.2314(2)	0.7629(4)	2.47(5)
C(23)	0.8682(2)	1.2082(2)	0.7950(4)	2.56(6)
C(24)	0.8163(2)	1.2779(2)	0.8033(4)	3.27(7)
C(25)	0.7034(3)	1.2523(2)	0.8380(5)	3.84(7)
C(26)	0.6427(3)	1.1577(2)	0.8622(4)	3.57(7)
C(27)	0.6926(2)	1.0843(2)	0.8561(4)	2.89(6)
C(28)	0.6330(3)	0.9859(2)	0.8842(4)	3.56(7)
C(29)	0.6831(3)	0.9163(2)	0.8811(5)	3.83(7)
C(30)	0.7960(3)	0.9406(2)	0.8471(4)	3.37(7)
C(31)	0.8567(2)	1.0361(2)	0.8162(4)	2.64(6)
C(32)	0.8063(2)	1.1094(2)	0.8224(4)	2.53(6)
C(33)	1.1624(2)	1.1923(2)	0.7440(4)	2.55(6)
C(34)	1.1983(2)	1.1478(2)	0.6139(4)	3.06(6)
C(35)	1.3140(3)	1.1775(2)	0.5991(5)	3.68(7)
C(36)	1.3939(3)	1.2508(2)	0.7148(5)	3.84(7)
C(37)	1.3593(3)	1.2947(2)	0.8469(5)	3.73(7)
C(38)	1.2436(2)	1.2659(2)	0.8624(4)	3.22(6)

TABLE III Intramolecular distances for hydroxyphenalenone moiety of **2**.

atom	atom	distance	atom	atom	distance
O(1)	C(1)	1.241(3)	O(3)	C(20)	1.242(3)
O(2)	C(3)	1.340(3)	O(4)	C(22)	1.346(3)
C(1)	C(2)	1.449(4)	C(20)	C(21)	1.450(4)
C(1)	C(12)	1.485(4)	C(20)	C(31)	1.482(4)
C(2)	C(3)	1.374(4)	C(21)	C(22)	1.365(4)
C(2)	C(14)	1.490(4)	C(21)	C(33)	1.494(4)
C(3)	C(4)	1.464(4)	C(22)	C(23)	1.458(4)
C(4)	C(5)	1.379(4)	C(23)	C(24)	1.381(4)
C(4)	C(13)	1.420(4)	C(23)	C(32)	1.422(4)
C(5)	C(6)	1.420(4)	C(24)	C(25)	1.409(4)
C(6)	C(7)	1.354(5)	C(25)	C(26)	1.363(4)
C(7)	C(8)	1.414(4)	C(26)	C(27)	1.409(4)
C(8)	C(9)	1.404(4)	C(27)	C(28)	1.413(4)
C(8)	C(13)	1.421(4)	C(27)	C(32)	1.416(4)
C(9)	C(10)	1.364(5)	C(28)	C(29)	1.359(5)
C(10)	C(11)	1.406(4)	C(29)	C(30)	1.408(4)
C(11)	C(12)	1.380(4)	C(30)	C(31)	1.387(4)
C(12)	C(13)	1.408(4)	C(31)	C(32)	1.412(4)

Distances are in angstroms. Estimated standard deviations in the least significant figure are given in parentheses.

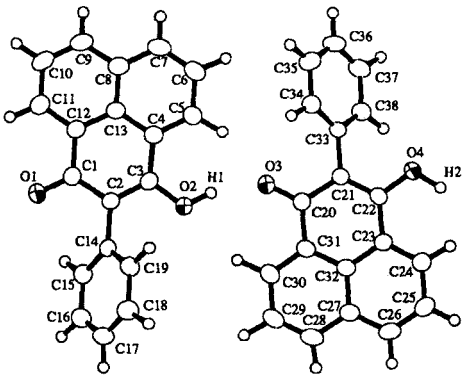
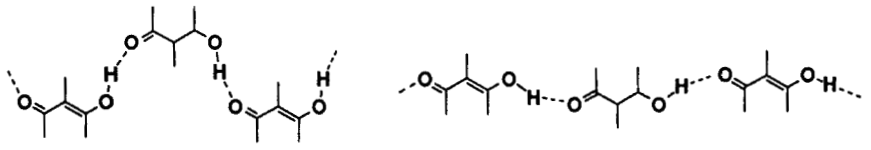


FIGURE 1 Molecular structure and atomic numbering scheme.

make a zigzag chain, which is the anti-anti configuration.[5] The H-bonding of 3-hydroxyphenalenone is reported to be the syn-syn[2]. Such a difference in the H-bonding configurations between both 3-hydroxyphenalenones with and without a phenyl group is the most characteristic feature in the crystal structures. The H-bonded molecules form a one dimensional ribbon along the *b* axis, which is stacked along the *c* axis. The average intermolecular distance between the slightly overlapped phenalenyl rings is 3.42 Å.



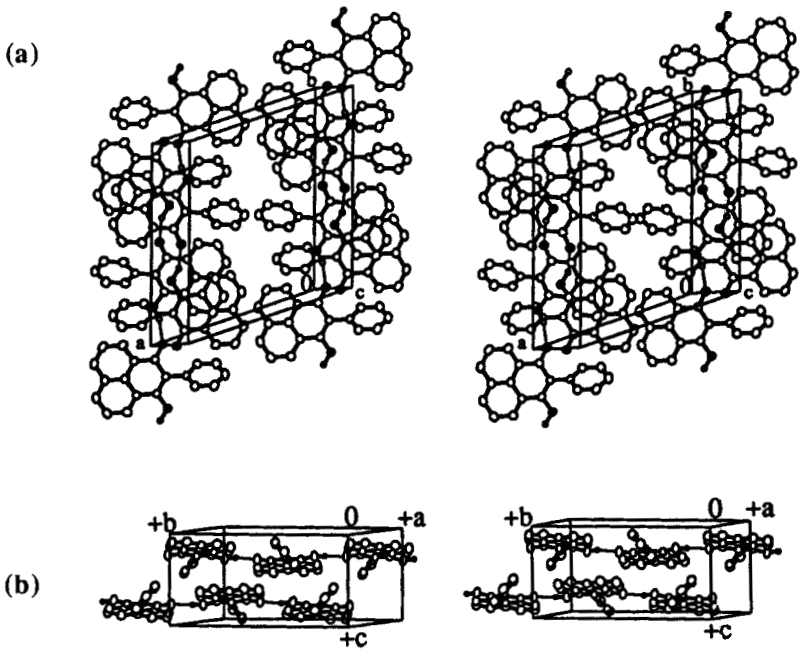


FIGURE 2 Stereo view of the crystal packing of **2** along (a) the *c* axis and (b) the *a* axis.

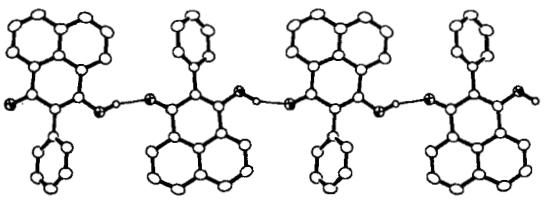


FIGURE 3 One dimensional linear tape arrangement of hydrogen-bonding in crystal structure of **2**.

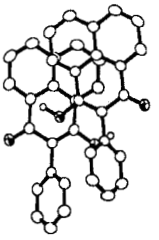


FIGURE 4 Overlap mode.

Strength of Intermolecular Hydrogen Bond

The infra-red spectrum of the molecule measured in KBr shows the broad O–H stretching vibration at 3204 cm^{-1}, while in a diluted solution of tetrachloroethylene the sharp absorption at 3509 cm^{-1} is observed. This feature is consistent with the existence of the O–H···O type H-bond in the crystalline state. The strength of the H-bonding can be conveniently estimated and classified into the three groups, weak, medium, and strong H-bond, according to the Novak classification using the O–H stretching frequency and the O···O distance[6]. The calculated value of the relative shift by the Novak procedure, 0.87% ((3509 – 3204)/3509), indicates that the molecule is classified into the weak H-bonded systems.

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