Only coordinates of H atoms refined and one overall $U_{\rm iso}$ parameter
Unit weights applied
$(\Delta/\sigma)_{\rm max} = 0.41$

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
O(1)	0.0921 (2)	0.6358 (1)	0.8014 (1)	0.0383
O(2)	0.3032 (3)	0.7306 (1)	0.8220 (2)	0.0598
C(1)	−0.0274 (3)	0.6624 (1)	0.7066 (2)	0.0344
C(2)	−0.1791 (3)	0.5997 (1)	0.6753 (2)	0.0301
C(3)	−0.2996 (4)	0.6335 (2)	0.5748 (2)	0.0386
C(4)	−0.3905 (4)	0.7077 (2)	0.5971 (2)	0.0455
C(5)	−0.2397 (4)	0.7658 (2)	0.6322 (2)	0.0475
C(6)	−0.1220 (4)	0.7775 (2)	0.5459 (2)	0.0483
C(7)	−0.0329 (4)	0.7034 (2)	0.5216 (2)	0.0439
C(8)	0.0893 (4)	0.6748 (2)	0.6194 (2)	0.0393
C(9)	−0.1173 (4)	0.7366 (1)	0.7301 (2)	0.0399
C(10)	−0.1845 (4)	0.6459 (2)	0.4870 (2)	0.0427
C(11)	−0.3057 (3)	0.5833 (1)	0.7600 (2)	0.0359
C(12)	−0.4675 (4)	0.5317 (2)	0.7140 (2)	0.0434
C(13)	−0.3919 (4)	0.4572 (2)	0.6814 (2)	0.0474
C(14)	−0.2886 (4)	0.4181 (2)	0.7778 (3)	0.0492
C(15)	−0.1273 (4)	0.4682 (2)	0.8270 (2)	0.0445
C(16)	0.0034 (4)	0.4813 (2)	0.7464 (2)	0.0416
C(17)	−0.0982 (4)	0.5217 (1)	0.6502 (2)	0.0381
C(18)	−0.2033 (4)	0.5434 (2)	0.8569 (2)	0.0381
C(19)	−0.2580 (4)	0.4691 (2)	0.6026 (2)	0.0456
C(20)	0.2436 (4)	0.6725 (2)	0.8500 (2)	0.0426
C(21)	0.3257 (5)	0.6300 (2)	0.9430 (2)	0.0520

Table 2. *Selected geometric parameters (Å, °)*

C(1)—C(2)	1.580 (3)	C(2)—C(11)	1.565 (3)
C(2)—C(3)	1.575 (3)	C(2)—C(17)	1.564 (3)
C(1)—C(2)—C(3)	102.7 (2)	C(1)—C(2)—C(17)	114.4 (2)
C(1)—C(2)—C(11)	114.4 (2)	C(3)—C(2)—C(17)	110.3 (2)
C(3)—C(2)—C(11)	109.9 (2)	C(11)—C(2)—C(17)	105.1 (2)
C(2)—C(1)—O(1)—C(20)	177.1 (2)		
C(1)—O(1)—C(20)—O(2)	−1.4 (2)		
C(1)—O(1)—C(20)—C(21)	178.9 (2)		

Table 3. *Contact distances (Å)*

O(1)···H(161)	2.26 (3)	O(2)···H(82)	2.36 (3)
O(1)···H(182)	2.29 (3)	O(2)···H(92)	2.51 (3)

H atoms were located by means of a Fourier difference map.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *CRYSTALS* (Watkin, Carruthers & Betteridge, 1988). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *CRYSTALS*. Molecular graphics: *CAMERON* (Pearce, Watkin & Prout, 1992). Software used to prepare material for publication: *CRYSTALS*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1240). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Boelema, E., Strating, J. & Wynberg, H. (1972). *Tetrahedron Lett.* pp. 1175–1178.

Enraf–Nonius (1989). *CAD-4 Software.* Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Graham, W. D. & Schleyer, P. von R. (1972). *Tetrahedron Lett.* pp. 1179–1180.

Graham, W. D., Schleyer, P. von R., Hageman, E. W. & Wenkert, E. (1973). *J. Am. Chem. Soc.* **95**, 5785–5786.

Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.

Lomas, J. S., Cordier, C. & Briand, S. (1996). *J. Chem. Soc. Perkin Trans.* 2, pp. 865–870.

Lomas, J. S., Cordier, C., Briand, S. & Vaissermann, J. (1996). *J. Chem. Soc. Perkin Trans.* 2, pp. 871–876.

Pearce, L., Watkin, D. J. & Prout, C. K. (1992). *CAMERON. Program for Plotting Molecular Structures.* Chemical Crystallography Laboratory, University of Oxford, England.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sosnowski, J. J., Rheingold, A. L. & Murray, R. K. (1985). *J. Org. Chem.* **50**, 3788–3791.

Watkin, D. J., Carruthers, J. R. & Betteridge, P. W. (1988). *CRYSTALS User Guide.* Chemical Crystallography Laboratory, University of Oxford, England.

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2,6-Dibenzoylhydroquinone

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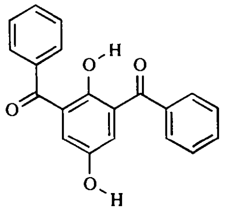
Abstract

The title compound, $C_{20}H_{14}O_4$, forms an infinite network of O—H···O and C—H···O hydrogen bonds. The phenyl rings of the benzoyl groups are non-coplanar with the hydroquinone moiety.

Comment

In the crystal structure of the title compound, (I), O—H···O and C—H···O hydrogen bonds (Desiraju, 1991) are found to be important.



(I)

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The two carbonyl groups point in roughly opposite directions. An *ORTEP*II (Johnson, 1976) plot of the structure is shown in Fig. 1. The two phenyl rings of the benzoyl groups are non-coplanar with the hydroquinone moiety, with the angles between the planes of the phenyl and hydroquinone rings being 55.3 and 63.7°. The hydrogen-bond network is shown in Fig. 2. The phenolic HO3 atom forms an intramolecular O—H···O hydrogen bond with the carbonyl O1 atom [H···O 1.74 (6), O···O 2.566 (3) Å, O—H···O 149 (5)°], while the other phenolic HO4 atom forms an intermolecular O—H···O hydrogen bond with the carbonyl O2^{i} atom [H···O^{i} 1.87 (4), O···O^{i} 2.715 (3) Å, O—H···O^{i} 170 (5)°; symmetry code: (i) $-\frac{1}{2}-x, 2-y, \frac{1}{2}+z$]. The carbonyl group which is involved in the intramolecular hydrogen bond is in the plane of the hydroquinone moiety [O1—C7—C8—C9 −9.0 (4)°], while the other carbonyl group is non-coplanar [O2—C14—C10—C9 137.1 (3)°]. The C7—O1 bond length [1.239 (3) Å] is somewhat longer than the C14—O2 bond length [1.225 (3) Å]. Similarly, the C9—O3 bond length [1.351 (3) Å] is shorter than the C12—O4 bond length [1.363 (3) Å]. These distance differences may be due to the formation of the six-membered intramolecular hydrogen bond between O3—H and O1=C7, which is similar to that observed in the crystal structure of naphthazarin (Shiau *et al.*, 1980). The phenolic O4^{ii} atom forms a C—H···O hydrogen bond with the C20—H group [H···O^{ii} 2.49 (3), C···O^{ii} 3.166 (4) Å, C—H···O^{ii} 132 (2)°; symmetry code: (ii) $-\frac{1}{2}-x, 2-y, z-\frac{1}{2}$] that is part of an infinite network of hydrogen bonds (Fig. 2). The other phenolic O atom, O3^{iii}, also participates in a C—H···O hydrogen bond with C19—H [H···O^{iii} 2.70 (5), C···O^{iii} 3.310 (4) Å, C—H···O^{iii} 122 (4)°; symmetry code: (iii) $\frac{1}{2}+x, \frac{3}{2}-y, 1-z$]. We note that the title compound adopts a non-centrosymmetric space group and that the relative orientation of the two phenolic H atoms is *syn*.

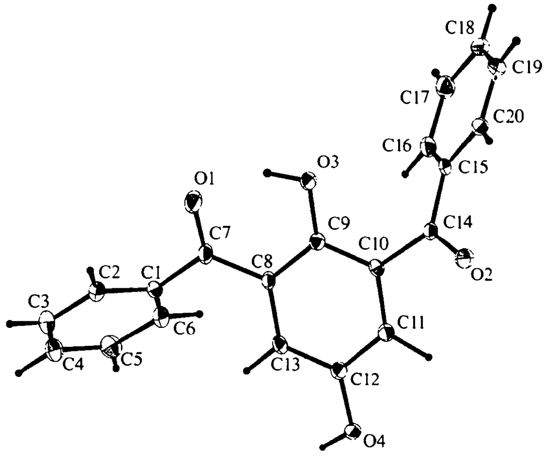

Fig. 1. An *ORTEP*II (Johnson, 1976) diagram of the title compound. Displacement ellipsoids are plotted at the 50% probability level and H atoms are drawn as small circles for clarity.

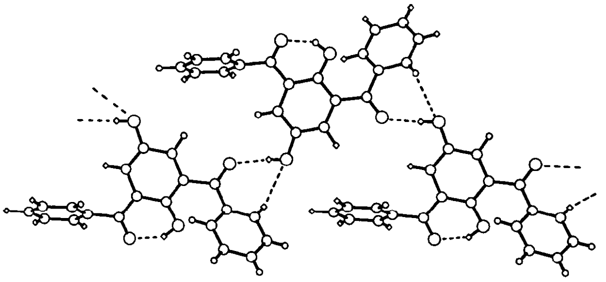

Fig. 2. The infinite network of O—H···O and C—H···O hydrogen bonds in the crystal structure of the title compound, with [001] horizontal and [010] vertical.

Experimental

2,6-Dibenzoylhydroquinone was prepared according to the literature procedure of Bogert & Howells (1930) and yellow crystals of the compound were obtained from ethanol solution.

Crystal data

$C_{20}H_{14}O_4$
$M_r = 318.31$
Orthorhombic
$P2_12_12_1$
$a = 8.0310\,(10)$ Å
$b = 13.1610\,(10)$ Å
$c = 14.4770\,(10)$ Å
$V = 1530.2\,(2)$ Å^3
$Z = 4$
$D_x = 1.382$ Mg m^{-3}
$D_m = 1.377$ Mg m^{-3}
D_m measured by flotation

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 250 reflections
$\theta = 10.5$–18.8°
$\mu = 0.096$ mm^{-1}
$T = 165\,(2)$ K
Needle
0.30 × 0.20 × 0.15 mm
Yellow

Data collection

Enraf–Nonius FAST area-detector diffractometer
Rotation scans
Absorption correction: none
2209 measured reflections
2209 independent reflections

2031 observed reflections [$I > 2\sigma(I)$]
$\theta_{max} = 28.69°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 18$
No standard reflections (area-detector data)

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0526$
$wR(F^2) = 0.1441$
$S = 0.711$
2207 reflections
273 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.1258P)^2 + 2.7021P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.105$

$\Delta\rho_{max} = 0.682$ e Å^{-3}
$\Delta\rho_{min} = -0.233$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = −0.3 (17)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
O1	0.0659 (3)	0.6626 (2)	0.87665 (15)	0.0282 (5)
O2	−0.1716 (3)	0.9239 (2)	0.54636 (13)	0.0196 (4)
O3	0.0722 (3)	0.74704 (15)	0.71695 (13)	0.0173 (4)
O4	−0.3226 (3)	1.0525 (2)	0.86010 (14)	0.0202 (4)
C1	−0.0128 (3)	0.7445 (2)	1.0138 (2)	0.0161 (5)
C2	−0.0683 (4)	0.6564 (2)	1.0590 (2)	0.0193 (5)
C3	−0.0801 (4)	0.6553 (2)	1.1549 (2)	0.0242 (6)
C4	−0.0346 (4)	0.7409 (3)	1.2060 (2)	0.0267 (7)
C5	0.0239 (4)	0.8271 (2)	1.1610 (2)	0.0238 (6)
C6	0.0333 (4)	0.8292 (2)	1.0649 (2)	0.0194 (6)
C7	0.0036 (4)	0.7399 (2)	0.9112 (2)	0.0176 (5)
C8	−0.0550 (3)	0.8242 (2)	0.8520 (2)	0.0134 (5)
C9	−0.0177 (3)	0.8223 (2)	0.7561 (2)	0.0132 (5)
C10	−0.0819 (3)	0.8991 (2)	0.6987 (2)	0.0130 (5)
C11	−0.1858 (3)	0.9739 (2)	0.7360 (2)	0.0150 (5)
C12	−0.2218 (4)	0.9762 (2)	0.8297 (2)	0.0148 (5)
C13	−0.1568 (3)	0.9014 (2)	0.8877 (2)	0.0146 (5)
C14	−0.0535 (3)	0.9023 (2)	0.5962 (2)	0.0143 (5)
C15	0.1158 (3)	0.8874 (2)	0.5574 (2)	0.0139 (5)
C16	0.2588 (4)	0.9103 (2)	0.6079 (2)	0.0180 (5)
C17	0.4154 (4)	0.9017 (2)	0.5680 (2)	0.0224 (6)
C18	0.4307 (4)	0.8694 (2)	0.4768 (2)	0.0231 (6)
C19	0.2885 (4)	0.8454 (2)	0.4260 (2)	0.0227 (6)
C20	0.1324 (4)	0.8546 (2)	0.4657 (2)	0.0171 (5)

Table 2. *Selected geometric parameters (Å, °)*

O1—C7	1.239 (3)	C8—C9	1.421 (3)
O2—C14	1.225 (3)	C9—C10	1.406 (3)
O3—C9	1.351 (3)	C10—C11	1.398 (4)
O4—C12	1.363 (3)	C10—C14	1.502 (3)
C1—C6	1.389 (4)	C11—C12	1.388 (4)
C7—C8	1.479 (4)	C12—C13	1.395 (4)
C8—C13	1.403 (4)	C14—C15	1.484 (4)
C6—C1—C2	120.0 (2)	C9—C10—C14	123.2 (2)
O1—C7—C8	120.7 (2)	C12—C11—C10	121.1 (2)
O1—C7—C1	118.1 (2)	O4—C12—C11	117.1 (2)
C8—C7—C1	121.2 (2)	O4—C12—C13	123.2 (2)
C13—C8—C9	119.7 (2)	C11—C12—C13	119.7 (2)
C13—C8—C7	121.1 (2)	C12—C13—C8	120.5 (2)
C9—C8—C7	119.1 (2)	O2—C14—C15	121.1 (2)
O3—C9—C10	118.4 (2)	O2—C14—C10	118.1 (2)
O3—C9—C8	122.4 (2)	C15—C14—C10	120.7 (2)
C10—C9—C8	119.1 (2)	C16—C15—C20	119.0 (3)
C11—C10—C9	119.8 (2)	C16—C15—C14	121.8 (2)
C11—C10—C14	116.9 (2)	C20—C15—C14	119.1 (2)

Data collection: *MADNES* (Enraf–Nonius, 1990). Cell refinement: *MADNES*. Data reduction: *MADNES*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PA1231). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bogert, M. T. & Howells, H. P. (1930). *J. Am. Chem. Soc.* **52**, 837–850.

Desiraju, G. R. (1991). *Acc. Chem. Res.* **24**, 290–296.

Enraf–Nonius (1990). *MADNES. Program for Area-Detector Data Interpretation.* Enraf–Nonius, Delft, The Netherlands.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO. Program for Plotting Molecular and Crystal Structures.* University of Cambridge, England.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Shiau, W.-I., Duesler, E. N., Paul, I. C., Curtin, D. Y., Blann, W. G. & Fyfe, C. A. (1980). *J. Am. Chem. Soc.* **102**, 4546–4548.

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Balancing Conjugational Stabilization and Torsional Strain. The Solid-State Structure of a 2-Thioalkyl-Substituted Pyridine *N*-Oxide

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Abstract

The solid-state geometry of 2-(1-phenyl-4-penten-1-ylthio)pyridine *N*-oxide, $C_{16}H_{17}NOS$, provides an insight into the spatial arrangement of the 2-thioalkyl side chain. The N2—C7—S8 bond angle [112.0 (2)°] shows a significant and unprecedented distortion from the expected value of 120° towards the pyridine *N*-oxide O atom. The substituents of the thioether are arranged to allow conjugational interaction of the lone pairs on sulfur and the heteroaromatic nucleus on one side, and a minimization of conformational strain between the pyridyl and the alkenyl groups on the other side.

Comment

Recent findings on the utility of *O*-alkylated 1-hydroxypyridine-2(1*H*)-thiones, *e.g.* (1), as alkoxyl radical pre-