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Inclusion compounds of 2,5-diphenylhydroquinone

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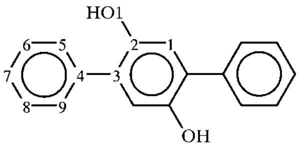
The crystal structures of three 1:2 inclusion compounds that consist of host molecule 2,5-diphenylhydroquinone ($C_{18}H_{14}O_2$) and the guest molecules 2-pyridone (C_5H_5NO), 1,3-diphenyl-2-propen-1-one (chalcone, $C_{15}H_{12}O$) and 1-(4-methoxyphenyl)-3-phenyl-2-propen-1-one (4′-methoxychalcone, $C_{16}H_{14}O_2$) were determined in order to study the ability of guest molecules in inclusion compounds to undergo photoreaction. All of the crystals were found to be photoresistant. The three inclusion compounds crystallize in triclinic space group $P\overline{1}$. In each case, the host/guest ratio is 1:2, with the host molecules occupying crystallographic centers of symmetry and the guest molecules occupying general positions. The guest molecules in each of the inclusion compounds are linked to the host molecules by hydrogen bonds. In the inclusion compound where the guest molecule is pyridone, the host molecule is disordered so that the hydroxy groups are distributed between two different sites, with occupancies of 0.738 (3) and 0.262 (3). The pyridone molecules form dimers *via* N—H⋯O hydrogen bonds.

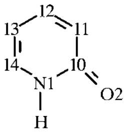
Comment

The crystal structures of the three inclusion compounds, *viz.* (I–*a*), (I–*b*) and (I–*c*) (see scheme below), were studied as part of a comprehensive investigation concerning the ability of guest molecules in inclusion compounds to undergo homogeneous photochemical reactions.

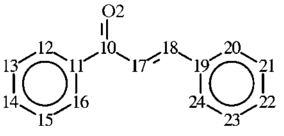
Inclusion compound (I–*a*) crystallizes in space group $P\overline{1}$. The host molecule occupies a crystallographic inversion center and the guest molecule, which is linked to the host *via* a hydrogen bond, occupies a general position. The host molecules are disordered so that the hydroxy groups occupy two different sites [with occupancies of 0.738 (3) and 0.262 (3), respectively] that are related by 180° rotation around the C7⋯C7($2 - x$, $1 - y$, $-z$) axis. The hydrogen bond in the major structure [Fig. 1 and Table 1; O1⋯O2 = 2.603 (2) Å] is shorter than that in the minor structure [Fig. 2; O1*A*⋯O2 = 2.973 (3) Å]. The O atom of the pyridone molecule forms a bifurcated hydrogen bond. In addition to the hydrogen bonds between the host and guest molecules, there are also hydrogen bonds between the guest molecules. These bonds involve atom N1 of one molecule and atom O2 of another molecule, and allow the formation of a dimer.



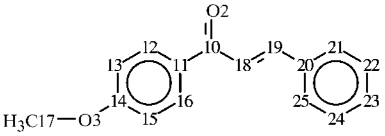
(I)



(*a*)



(*b*)



(*c*)

Compound (I–*b*) crystallizes in space group $P\overline{1}$. The host molecule occupies a crystallographic inversion center and the guest molecule, which is linked to the host *via* a hydrogen bond, occupies a general position (Fig. 3 and Table 1). The degree of planarity of the 1,3-diphenyl-2-propen-1-one molecule [chalcone, (*b*)] in (I–*b*) differs from the conformation in

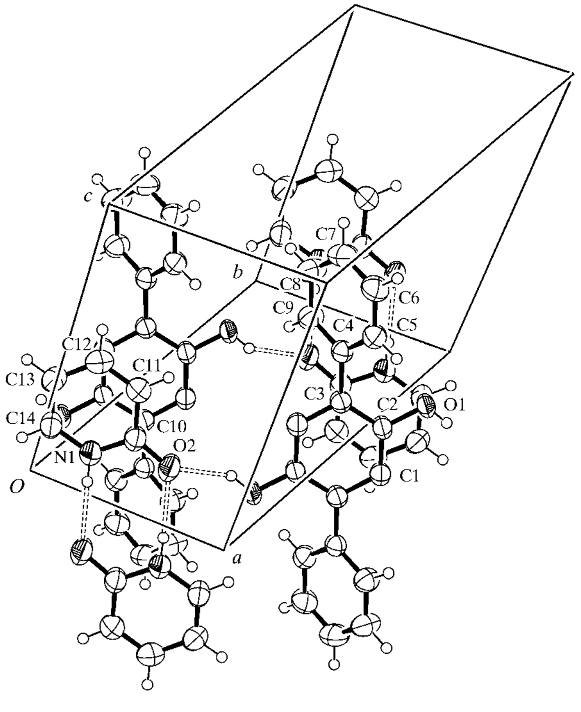


Figure 1
A view of the packing of molecules in (I–*a*), showing the hydrogen bonding between the host and guest molecules in the major structure and between the pyridone molecules. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

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the two polymorphs of pure (*b*) [Rabinovich (1970), refcode BZYACO in the Cambridge Structural Database (Allen, 2002); Ohkura *et al.* (1973), refcode BZYACO01] and from that of (*b*) in another inclusion compound [Kaftory *et al.* (1985), refcode DEGPUA]. A comparison of the C11—C10—C17—C18 and C10—C17—C18—C19 torsion angles in the above structures is given in Table 2. It can be seen that the C11—C10—C17—C18 torsion angle in (I–*b*) is the smallest.

Compound (I–*c*) crystallizes in space group $P\overline{1}$. Two guest molecules (labeled *A* and *B*) occupy general positions, while two host molecules (labeled *A* and *B*) occupy different crystallographic inversion centers. The guest molecules are linked to the host molecules *via* hydrogen bonds (see Table 1). The *A* and *B* host–guest couples are packed orthogonally to one another, as can be seen in Fig. 4. The geometry of 4′-methoxychalcone, (*c*), in (I–*c*) is similar to the geometry of pure (*c*) [Li *et al.* (1992), refcode KOTSER]. The molecule of (*c*) in (I–*c*) is almost planar. A comparison of the torsion angles is given in Table 2.

In each compound, the conformation of the host molecule can be defined by two dihedral angles around the single bonds. However, since the molecule occupies a crystallographic inversion center in all known compounds that include 2,5-diphenylhydroquinone [the present work; Kaftory *et al.* (1985), refcode DEGREI], the conformation is described by only one dihedral angle. The dihedral angles between the two phenyl rings are 13.9 (1)° for the host molecule in (I–*a*), 20.1 (1)° for the host molecule in (I–*b*), and 7.0 (2) and

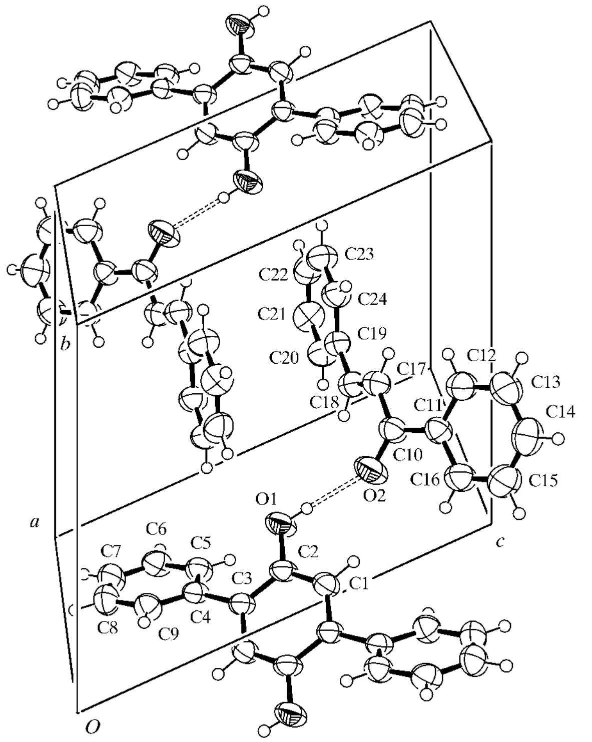

Figure 3
A view of the packing of molecules in (I–*b*), showing the hydrogen bonding between the host and guest molecules. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

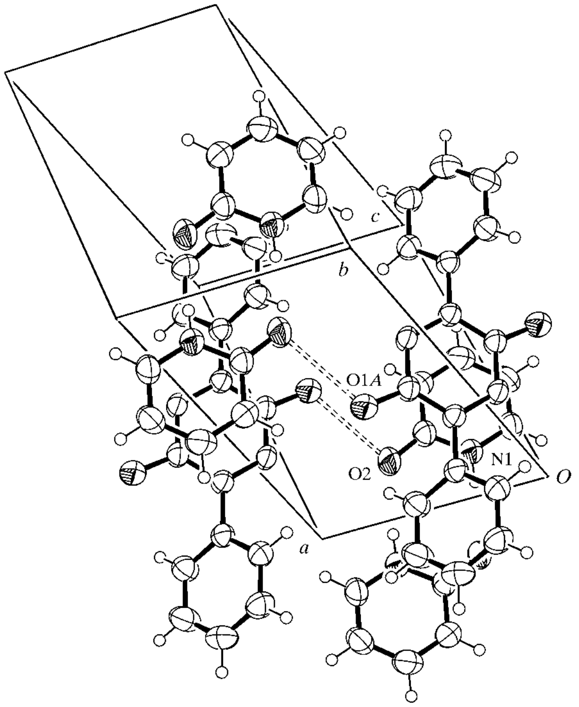

Figure 2
A view of the packing of molecules in (I–*a*), showing the hydrogen bonding between the host and guest molecules in the minor structure and between the pyridone molecules. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

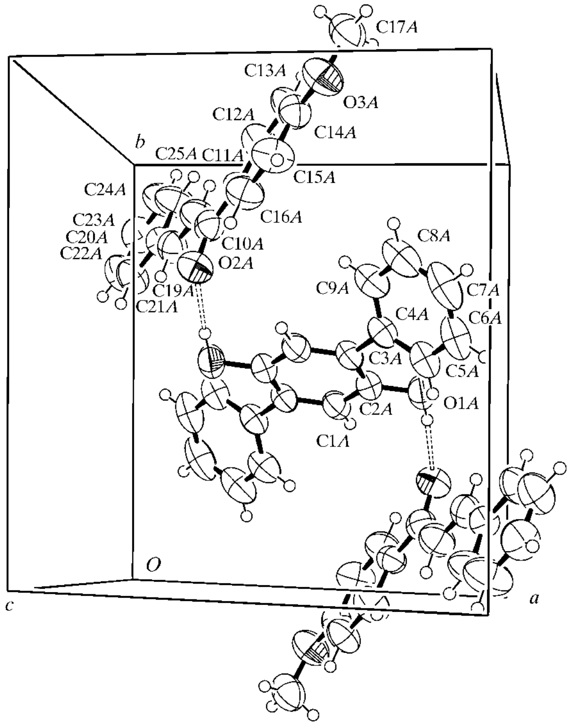

Figure 4
A view of the packing of molecules in (I–*c*), showing the hydrogen bonding between the host and guest molecules. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

58.8 (1)° for the two host molecules in the asymmetric unit of (I–*c*). The dihedral angle between these planes in the inclusion compound with dibenzylideneacetone [Kaftory *et al.* (1985), refcode DEGREI] is 24.3 (2)°. The distances between potentially photoreactive centers are too long to enable photodimerization in the solid state. The shortest distance between two potentially reactive atoms of the guest molecules that are related by an inversion center [C11···C14($-x$, $-y$, $1-z$)] in (I–*a*) is 5.691 (3) Å, and the shortest distance in (I–*b*) is 6.201 (4) Å [C17···C18($-x$, $1-y$, $1-z$)]. The distances between potentially reacting atoms in guest molecules *A* [C18···C19($-x$, $-y$, $-z$)] and *B* [C18···C19($-x$, $1-y$, $1-z$)] in (I–*c*) are 4.701 (6) and 4.333 (6) Å, respectively. These distances are larger than the limit set for potential photochemical reactions (Cohen & Schmidt, 1964). The relationship between the two guest molecules in (I–*c*) is shown in Fig. 5.

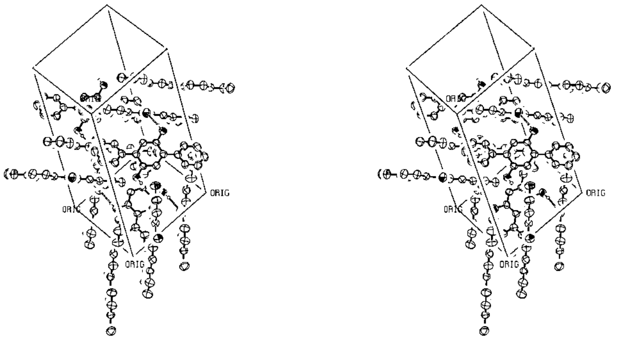

Figure 5
A view of the packing of molecules in (I–*c*), showing the orthogonal arrangement. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

Experimental

The host component was synthesized according to the method of Toda *et al.* (1989). The purification of 2,5-diphenylhydroquinone was checked by comparing its ^{1}H NMR spectrum and melting-point value to those reported in the literature (Ohba *et al.*, 1997). The guest components in all three inclusion compounds were purchased from Sigma. Crystals of the inclusion compounds were obtained from ethyl acetate solutions of the host and guest in a 1:2 ratio by slow evaporation of the solvent at room temperature.

Compound (I–*a*)

Crystal data

$C_{18}H_{14}O_2 \cdot 2C_5H_5NO$
M_r = 452.49
Triclinic, $P\overline{1}$
a = 6.510 (1) Å
b = 8.971 (1) Å
c = 11.085 (2) Å
α = 70.746 (2)°
β = 73.108 (2)°
γ = 73.807 (2)°
V = 572.66 (15) Å^3
Z = 1
D_x = 1.312 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2021 reflections
θ = 2.5–25.0°
μ = 0.09 mm^{-1}
T = 293 (2) K
Prism, colorless
0.3 × 0.2 × 0.1 mm

Data collection

Nonius KappaCCD diffractometer
φ scans
2021 measured reflections
2021 independent reflections
1611 reflections with $I > 2\sigma(I)$
θ_{max} = 25.0°
$h = 0 \rightarrow 7$
$k = -9 \rightarrow 10$
$l = -12 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.126
S = 1.06
2021 reflections
165 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.07P)^2 + 0.0744P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

Compound (I–*b*)

Crystal data

$C_{18}H_{14}O_2 \cdot 2C_{15}H_{12}O$
M_r = 678.78
Triclinic, $P\overline{1}$
a = 9.413 (2) Å
b = 10.042 (2) Å
c = 11.848 (2) Å
α = 65.74 (2)°
β = 81.57 (2)°
γ = 62.81 (2)°
V = 907.0 (3) Å^3
Z = 1
D_x = 1.243 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2649 reflections
θ = 1.0–25.1°
μ = 0.08 mm^{-1}
T = 293 (2) K
Prism, yellow
0.20 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
φ scans
5064 measured reflections
3153 independent reflections
1284 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 25.1°
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.142
S = 0.86
3153 reflections
239 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.06P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Compound (I–*c*)

Crystal data

$C_{18}H_{14}O_2 \cdot 2C_{16}H_{14}O_2$
M_r = 738.84
Triclinic, $P\overline{1}$
a = 10.322 (2) Å
b = 11.051 (2) Å
c = 18.041 (3) Å
α = 83.11 (2)°
β = 75.54 (2)°
γ = 89.70 (2)°
V = 1977.6 (7) Å^3
Z = 2
D_x = 1.241 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 6451 reflections
θ = 1.0–25.4°
μ = 0.08 mm^{-1}
T = 293 (2) K
Prism, yellow
0.20 × 0.10 × 0.08 mm

Data collection

Nonius KappaCCD diffractometer
φ scans
6990 measured reflections
6990 independent reflections
2911 reflections with $I > 2\sigma(I)$
θ_{max} = 25.4°
$h = 0 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = -20 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.224$
$S = 0.85$
6990 reflections
506 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1252P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.14$ e Å^{-3}
$\Delta\rho_{\min} = -0.14$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °) in the three title compounds.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
Compound (I–*a*)				
O1—H1O1···O2$^{\text{i}}$	0.82	1.86	2.603 (2)	151
N1—H1N···O2$^{\text{ii}}$	0.86	2.00	2.854 (2)	171
Compound (I–*b*)				
O1—H1O1···O2	0.82	1.98	2.803 (3)	177
Compound (I–*c*)				
O1*A*—H1O*A*···O2*A*$^{\text{iii}}$	0.82	1.98	2.793 (4)	172
O1*B*—H1O*B*···O2*B*$^{\text{iv}}$	0.82	1.91	2.727 (4)	174

Symmetry codes: (i) $2 - x, 1 - y, -z$; (ii) $1 - x, -y, -z$; (iii) $1 - x, 1 - y, -z$; (iv) $x, y - 1, z$.

Table 2
Comparison of selected torsion angles (°) in calcone and 4′-methoxychalcone.

Chalcone	C11—C10—C17—C18	C10—C17—C18—C19
(I–*b*)	153.3	175.3
DEGPUA	171.9	−179.4
BZYACO	−160.5	178.7
BZYACO01	167.8	177.8
4′-Methoxychalcone	C11—C10—C18—C19	C10—C18—C19—C20
(I–*c*), molecule *A*	−177.6	−179.0
(I–*c*), molecule *B*	−179.0	−179.5
KOTSER	160.4	176.4

In all three structures, hydroxy H atoms were located from difference Fourier maps and treated as riding. During the refinement of (I–*a*), the occupancies of the two hydroxy O atoms were free to refine. The H atom of the major O atom was located from a difference Fourier map; however, the minor O atom could not be detected.

For all three compounds, data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV1153). Services for accessing these data are described at the back of the journal.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Kaftory, M., Tanaka, K. & Toda, F. (1985). *J. Org. Chem.* **50**, 2154–2158.
Li, Z., Huang, L. & Su, G. (1992). *Acta Cryst.* C**48**, 751–752.
Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.
Ohba, Y., Murakami, Y., Sone, T. & Awano, H. (1997). *J. Heterocycl. Chem.* **34**, 781–787.
Ohkura, K., Kashino, S. & Haisa, M. (1973). *Bull. Chem. Soc. Jpn*, **46**, 627–628.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Rabinovich, D. (1970). *J. Chem. Soc. B*, pp. 11–16.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Toda, F., Tanaka, K. & Tange, H. (1989). *J. Chem. Soc. Perkin Trans.* 1, pp. 1555–1556.