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Structural effects on the solid-state photodimerization of 2-pyridone derivatives in inclusion compounds

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The structures of six crystalline inclusion compounds between various host molecules and three guest molecules based on the 2-pyridone skeleton are described. The six compounds are 1,1′-biphenyl-2,2′-dicarboxylic acid–2-pyridone (1/2), $C_{14}H_{10}O_4\cdot2C_5H_5NO$, (I–*a*), 1,1′-biphenyl-2,2′-dicarboxylic acid–4-methyl-2-pyridone (1/2), $C_{14}H_{10}O_4\cdot2C_6H_7NO$, (I–*c*), 1,1′-biphenyl-2,2′-dicarboxylic acid–6-methyl-2-pyridone (1/2), $C_{14}H_{10}O_4\cdot2C_6H_7NO$, (I–*d*), 1,1,6,6-tetraphenyl-2,4-hexadiyne-1,6-diol–1-methyl-2-pyridone (1/2), $C_{30}H_{22}O_2\cdot2C_6H_7NO$, (II–*b*), 1,1,6,6-tetraphenyl-2,4-hexadiyne-1,6-diol–4-methy-2-pyridone (1/2), $C_{30}H_{22}O_2\cdot2C_6H_7NO$, (II–*c*), and 4,4′,4′′-(ethane-1,1,1-triyl)triphenol–6-methyl-2-pyridone–water (1/3/1), $C_{20}H_{18}O_3\cdot3C_6H_7NO\cdot H_2O$, (III–*d*). In two of the compounds, (I–*a*) and (I–*d*), the host molecules lie about crystallographic twofold axes. In two other compounds, (II–*b*) and (II–*c*), the host molecules lie across inversion centers. In all cases, the guest molecules are hydrogen bonded to the host molecules through O—H···O═C hydrogen bonds [the range of O···O distances is 2.543 (2)–2.843 (2) Å. The pyridone moieties form dimers through N—H···O═C hydrogen bonds in five of the compounds [the range of N···O distances is 2.763 (2)–2.968 (2) Å]. In four compounds, (I–*a*), (I–*c*), (I–*d*) and (II–*c*), the molecules are arranged in extended zigzag chains formed *via* host–guest hydrogen bonding. In five of the compounds, the guest molecules are arranged in parallel pairs on top of each other, related by inversion centers. However, none of these compounds underwent photodimerization in the solid state upon irradiation. In one of the crystalline compounds, (III–*d*), the guest molecules are arranged in stacks with one disordered molecule. The unsuccessful dimerization is attributed to the large interatomic distances between the potentially reactive atoms [the range of distances is 4.027 (4)–4.865 (4) Å] and to the bad overlap, expressed by the lateral shift between the orbitals of these atoms [the range of the shifts from perfect overlap is 1.727 (4)–3.324 (4) Å]. The bad overlap and large distances between potentially photoreactive atoms are attributed to the hydrogen-bonding schemes, because the interactions involved in hydrogen bonding are stronger than those in π–π interactions.

Comment

Inducing photochemical reactions in inclusion compounds has proved to be a unique method for synthesizing a large variety of compounds (Tanaka & Toda, 2002). Understanding the mechanism and geometric requirements needed to enable such reactions depend on our knowledge of the molecular structure and the arrangement of molecules in the crystal. It would be an advantage to be able to monitor structural changes at different stages of the reaction. However, in most cases, the crystal breaks and its crystal structure cannot be determined. Nevertheless, there are more than a few examples of such reactions where the crystal integrity is retained throughout the reaction (homogeneous photochemical reaction) (Wegner, 1969; Osaki & Schmidt, 1972; Cheng & Foxman, 1977; Nakanishi *et al.*, 1981; Chang *et al.*, 1982; Ohashi *et al.*, 1982; Braun & Wegner, 1983; Tieke & Chapuis, 1984; Wang & Jones, 1987; Leibovitch *et al.*, 1998). In some cases, the crystal structures of a solid solution containing both the reactant and the product were analyzed structurally (Nakanishi *et al.*, 1981; Chang *et al.*, 1982; Leibovitch *et al.*, 1998; Theocharis & Desiraju, 1984; Turowska-Tyrk, 2003; Turowska-Tyrk & Trzop, 2003; Zouev *et al.*, 2006; Lavy & Kaftory, 2007; Lavy *et al.*, 2008). In a neat solid photoreactive compound, the molecular structural changes induced by the reaction affect and interfere with the neighboring molecules. However, the same molecule in inclusion compounds is surrounded by host molecules that are not involved in the reaction and are thus not expected to undergo structural changes. Therefore, the volume available for the guest molecule to accommodate its structural change determines the homogeneity of the reaction. This volume is also called the 'reaction cavity', a concept that was originally introduced and developed by Cohen (1975) to describe reactions in crystals. This model was further developed by Weiss *et al.* (1993) and

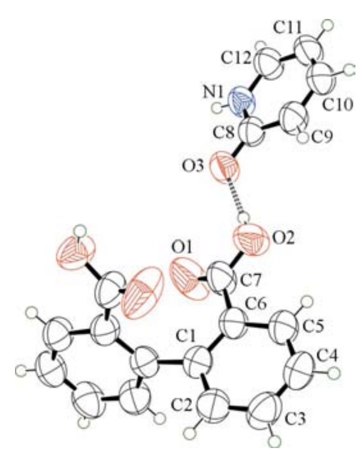

Figure 1
The molecular structure of (I–*a*). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

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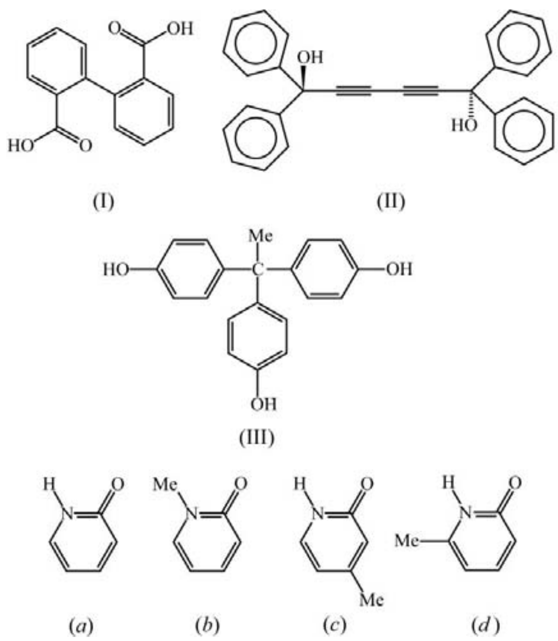
Keating & Garcia-Garibay (1998). The ($4\pi s + 4\pi s$) photocycloadditions are among the oldest known and, together with the ($2\pi s + 2\pi s$) cycloadditions, constitute an important group of photochemical reactions. The demonstration that photodimerization of pyridone is homogeneous throughout the entire reaction (Lavy *et al.*, 2008) prompted us to examine similar systems. However, these systems do not exhibit photodimerization. We present here the structures of six inclusion compounds and discuss the failure of the systems to undergo solid-state photodimerization.



The six inclusion compounds are 1,1′-biphenyl-2,2′-dicarboxylic acid–2-pyridone (1/2), (I–*a*) (Fig. 1), 1,1′-biphenyl-2,2′-dicarboxylic acid–4-methyl-2-pyridone (1/2), (I–*c*) (Fig. 2), 1,1′-biphenyl-2,2′-dicarboxylic acid–6-methyl-2-pyridone (1/2), (I–*d*) (Fig. 3), 1,1,6,6-tetraphenyl-2,4-hexadiyne-1,6-diol–1-methyl-pyridone (1/2), (II–*b*) (Fig. 4), 1,1,6,6-tetraphenyl-2,4-hexadiyne-1,6-diol–4-methyl-2-pyridone (1/2), (II–*c*) (Fig. 5), and 4,4′,4′′-(ethane-1,1,1-triyl)triphenol–6-methyl-2-pyridone–water (1/3/1), (III–*d*) (Fig. 6). 1,1′-Biphenyl-2,2′-dicarboxylic acid, (I), has the trivial name diphenic acid. The host molecules in (I–*a*) and (I–*d*) lie on twofold symmetry axes, while those in (II-*b*) and (II-*c*) straddle inversion centres. There are two crystallographic independent guest molecules in (I–*c*). In (III–*d*), there are three guest molecules in the asymmetric unit for each host molecule, as well as a water molecule: two of the guest molecules are ordered, while the third is disordered over two sites related by a rotation axis (see *Experimental*). In all six compounds, the guest molecules, (*a*)–(*d*) (see scheme), are hydrogen bonded to the host molecules, (I)–(III), through

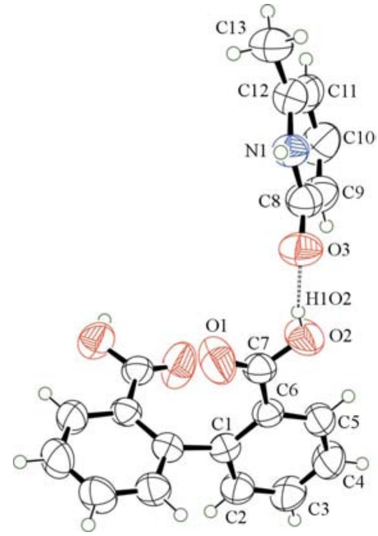


Figure 3
The molecular structure of (I–*d*). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

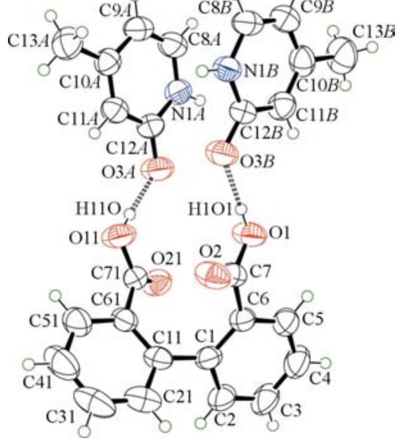


Figure 2
The molecular structure of (I–*c*). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

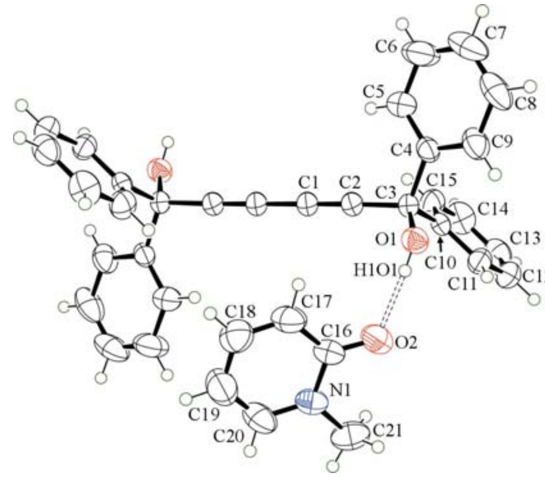


Figure 4
The molecular structure of (II–*b*). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

C═O···H—O interactions [the range of O···O distances is 2.543 (2)–2.843 (2) Å; see Table 1 and Figs. 1–6]. In five of the compounds, the guest molecules [(*a*), (*c*) and (*d*)] form dimers by hydrogen bonds of the C═O···H—N type through inversion centers. The exception is guest molecule (*b*), in which the hydrogen-bond donor (N—H) was replaced by N—Me. Such hydrogen-bonding schemes that form dimers are typical of pyridone-like compounds possessing H—N—C═O units. The arrangement of the guest and host molecules is determined by the hydrogen-bonding schemes. The packing of molecules in the unit cell showing the different schemes of hydrogen bonds together with the mutual geometric relations between pairs of guest molecules are shown in Figs. 7–12. In (I–*a*), (I–*c*) and (I–*d*) (Figs. 7–9), each of the host molecules is hydrogen bonded through its hydroxy groups to two guest molecules that form dimers through hydrogen bonds. The molecules are arranged in extended zigzag chains. A similar arrangement is observed in (II–*c*) (Fig. 11). In the absence of a hydrogen-bond donor (N—H) in the guest of (II–*b*), the dimers are not formed and therefore the chain is replaced by isolated host molecules hydrogen bonded to two guest molecules (Fig. 10). The packing of molecules in (III–*d*) is different (Fig. 12) as a consequence of the presence of three hydrogen-bond donors in the host molecule, and the presence of a water molecule. The latter serves as mediator for hydrogen bonding between two host molecules and a guest. One of the guest molecules does not participate in the hydrogen-bond schemes and its role is a space-filling one. The space available for this molecule is large and the molecule is accommodated in disordered manner.

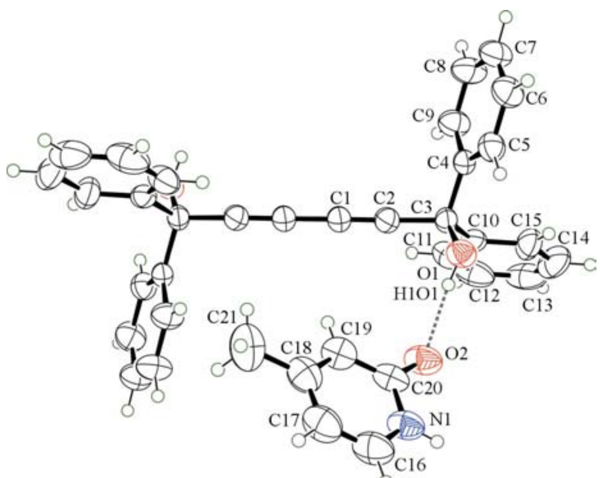


Figure 5
The molecular structure of (II–*c*). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

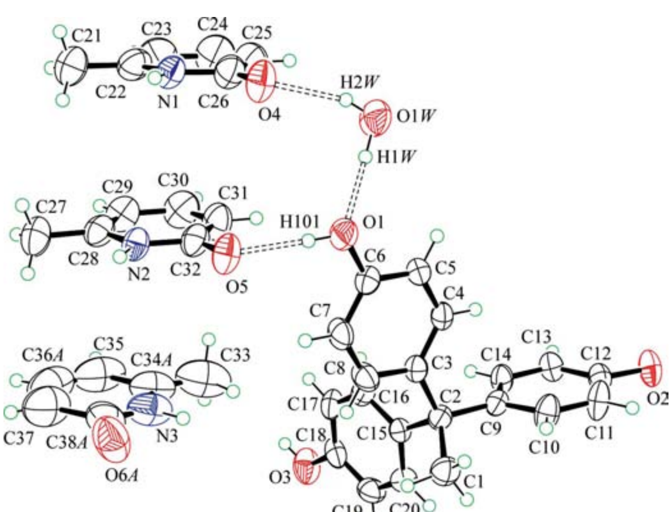


Figure 6
The molecular structure of (III–*d*). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. For clarity, the minor component of the disordered pyridone guest molecule has been omitted.

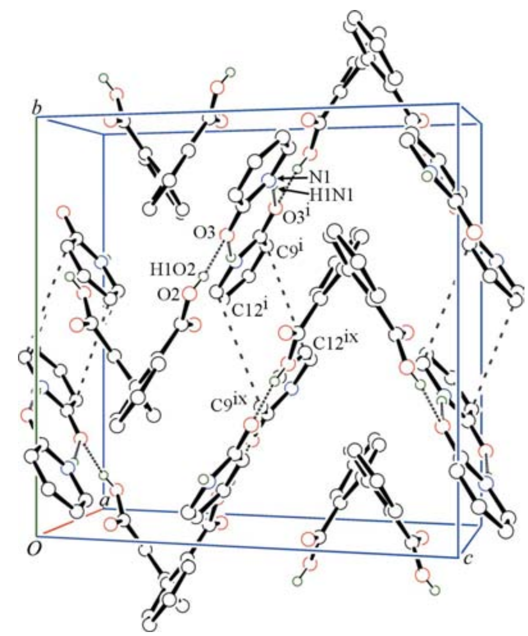


Figure 7
The packing of molecules in the unit cell, showing also the distances between potentially reactive centers in (I–*a*). [Symmetry codes: (i) $-x + \frac{1}{2}$, $-y + \frac{3}{2}$, $-z + 1$; (ix) $x + \frac{1}{2}$, $y - \frac{1}{2}$, z.]

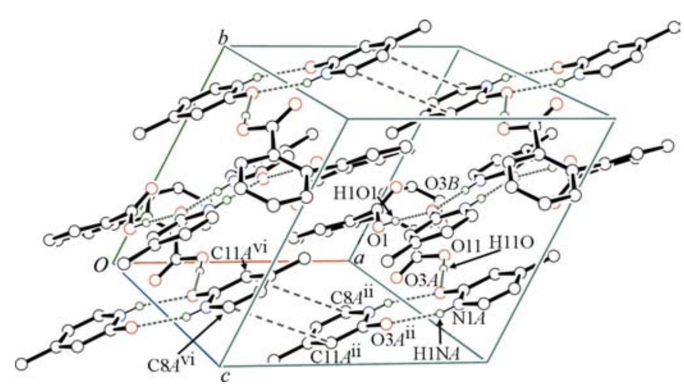


Figure 8
The packing of molecules in the unit cell, showing also the distances between potentially reactive centers in (I–*c*). [Symmetry codes: (ii) $-x + 2$, $-y$, $-z + 1$; (vi) $x - 1$, y, z.]

A search of the Cambridge Structural Database (Allen, 2002) provided 135 compounds containing the pyridone skeleton. The average C═O distance from 169 hits is 1.26 (2) Å, slightly longer than the C═O carbonyl bonds that are not involved in hydrogen bonding. The average intermolecular O···N distance is 2.80 (5) Å, and the average N—H···O angle is 170 (7)°. The ranges of the corresponding parameters in the compounds presented here are 1.247 (2)–1.288 (5) Å, 2.763 (2)–2.968 (2) Å and 163–178°, respectively.

The phenomenon of [4+4] photodimerization in the solid state is highly dependent on the mutual arrangement of the two monomers, on the distances between the reactive centers and on the substituents carried by the monomer. In the ideal case, the substituents are very small (normally H atoms), the double bonds are parallel, the orbitals of the reacting centers are overlapping and the distances between the centers are 3.5–4.2 Å. In cases where these requirements are not met, the photoreaction will fail to proceed. The potentially reactive compounds (*a*)–(*d*) do not have bulky groups as substituents and therefore it was expected that the other requirements would be fulfilled. It turned out that none of the inclusion compounds was photoactive. The geometric relationships

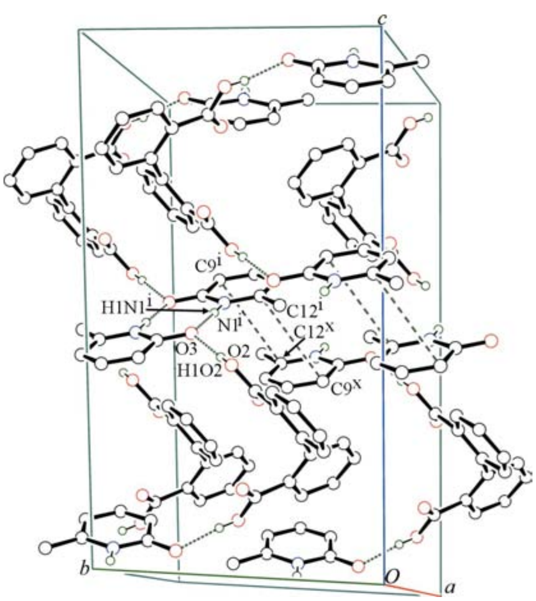


Figure 9
The packing of molecules in the unit cell, showing also the distances between potentially reactive centers in (I–*d*). [Symmetry codes: (i) $-x+\frac{1}{2}$, $-y+\frac{3}{2}$, $-z+1$; (x) $x+\frac{1}{2}$, $-y-\frac{1}{2}$, z.]

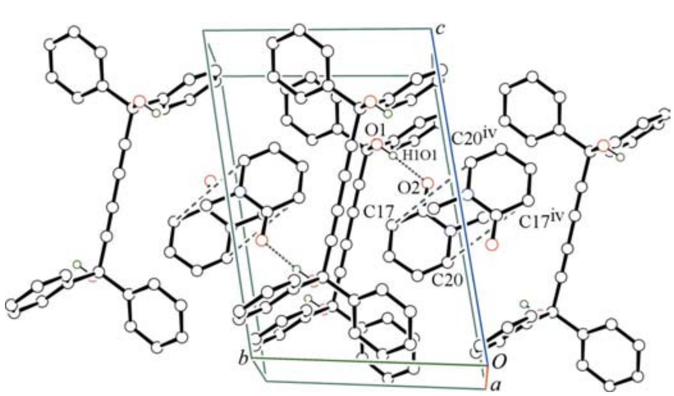


Figure 10
The packing of molecules in the unit cell, showing also the distances between potentially reactive centers in (II–*b*). [Symmetry code: (iv) $-x+1$, $-y$, $-z+1$.]

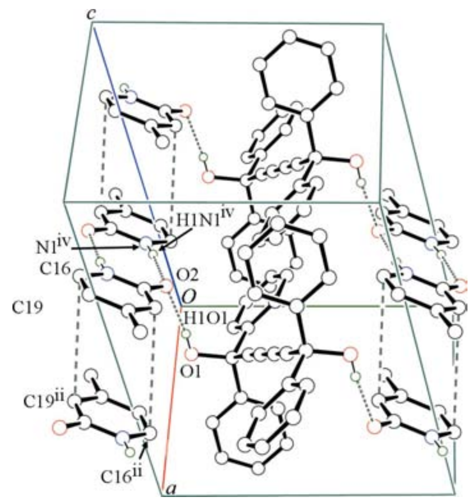


Figure 11
The packing of molecules in the unit cell, showing also the distances between potentially reactive centers in (II–*c*). [Symmetry codes: (ii) $-x+2$, $-y$, $-z+1$; (iv) $-x+1$, $-y$, $-z+1$.]

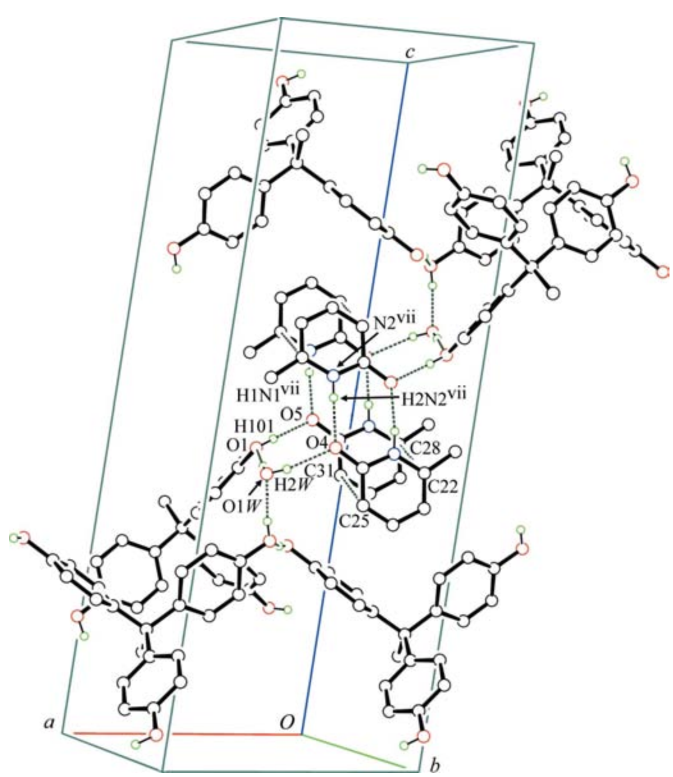


Figure 12
The packing of molecules in the unit cell, showing also the distances between potentially reactive centers in (III–*d*), omitting the disordered molecule. [Symmetry code: (vii) $-x+1$, $-y+2$, $-z+1$.]

between the guest molecules is summarized in Table 2 and each of the compounds fails to meet one of the requirements. In (I–*c*) and (II–*b*), the distances between the reacting centers [4.865 (4) and 4.769 (4) Å, respectively] are above the limit (4.2 Å) set by Schmidt (1971). In (I–*a*), (I–*c*), (I–*d*), (II–*b*) and (II–*c*), the lateral shifts between the orbitals are too large [2.257 (4), 3.324 (4), 1.929 (6), 3.122 (4) and 1.996 (5) Å, respectively] to allow the overlap needed for the reaction to take place (Ramamurthy & Venkatesan, 1987; Zolotoy *et al.*, 2002). Compound (III–*d*) shows the best geometry between the guest molecules, such as the shortest distances between reactive atoms and the shortest lateral shift of the orbitals; nevertheless, irradiation did not reveal the expected results. This behavior might be attributed to the mutual orientation, namely head-to-head with the methyl groups overlapping each other. It is important to note, however, that irradiation of solid inclusion compounds of diphenic acid with 5-chloro- or 5-methyl-2-pyridone revealed [2+2] photodimerization to the corresponding *cis–anti* dimer (Hirano *et al.*, 2005). However, in the later, the distances between the reacting atoms were very short (3.458 and 3.458 Å) and the methyl groups did not overlap each other. It was expected that the packing would be governed by the π–π interactions between guest molecules, which would determine the mutual geometry enabling photodimerization. However, the stronger intermolecular interactions of hydrogen bonds prevailed and determined the molecular packing. We therefore attribute the geometric relations between the guest molecules to the hydrogen-bonding interactions.

Experimental

Commercially available reagents were purchased from Aldrich and used without further purification. All inclusion compounds were prepared by mixing stoichiometric amounts of the host and guest compounds in ethyl acetate, followed by slow evaporation to yield crystals of the inclusion compounds.

Compound (I–*a*)

Crystal data

$C_{14}H_{10}O_4 \cdot 2C_5H_5NO$
M_r = 432.42
Monoclinic, $C2/c$
a = 10.569 (1) Å
b = 14.054 (3) Å
c = 15.016 (1) Å
β = 105.95 (3)°
V = 2144.6 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.20 × 0.10 × 0.04 mm

Data collection

Nonius KappaCCD diffractometer
6780 measured reflections
2013 independent reflections
1489 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.200
S = 1.03
2013 reflections
157 parameters
Only H-atom displacement parameters refined
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Compound (I–*c*)

Crystal data

$C_{14}H_{10}O_4 \cdot 2C_6H_7NO$
M_r = 460.47
Triclinic, $P\overline{1}$
a = 9.838 (2) Å
b = 10.085 (2) Å
c = 14.016 (3) Å
α = 89.77 (3)°
β = 74.90 (3)°
γ = 62.764 (2)°
V = 1183.0 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.20 × 0.20 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
9471 measured reflections
3998 independent reflections
3051 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.141
S = 0.91
3998 reflections
307 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Compound (I–*d*)

Crystal data

$C_{14}H_{10}O_4 \cdot 2C_6H_7NO$
M_r = 460.47
Monoclinic, $C2/c$
a = 11.415 (2) Å
b = 10.957 (2) Å
c = 19.660 (3) Å
β = 106.59 (2)°
V = 2356.6 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.30 × 0.25 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
8025 measured reflections
2063 independent reflections
1408 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.078
$wR(F^2)$ = 0.287
S = 1.09
2063 reflections
158 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Compound (II–*b*)

Crystal data

$C_{30}H_{22}O_2 \cdot 2C_6H_7NO$
M_r = 632.73
Triclinic, $P\overline{1}$
a = 7.305 (1) Å
b = 9.369 (2) Å
c = 13.292 (3) Å
α = 77.32 (2)°
β = 89.46 (2)°
γ = 76.57 (3)°
V = 862.4 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.08 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
8783 measured reflections
3182 independent reflections
2398 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.135$
$S = 0.98$
3182 reflections
220 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

Compound (II–*c*)

Crystal data

$C_{30}H_{22}O_2 \cdot 2C_6H_7NO$
$M_r = 632.73$
Triclinic, $P\overline{1}$
$a = 8.640$ (2) Å
$b = 10.203$ (2) Å
$c = 11.403$ (3) Å
$\alpha = 106.71$ (3)°
$\beta = 111.54$ (2)°
$\gamma = 95.87$ (2)°
$V = 870.8$ (4) Å^3
$Z = 1$
Mo $K\alpha$ radiation
$\mu = 0.08$ mm^{-1}
$T = 293$ K
0.25 × 0.10 × 0.05 mm

Table 1
Hydrogen-bond geometry (Å, °).

Compound	D—H···A	D—H	H···A	D···A	D—H···A
(I–*a*)	O2—H1O2···O3	0.82	1.76	2.579 (2)	172
	N1—H1N1···O3^{i}	0.86	2.07	2.916 (2)	168
(I–*c*)	O1—H1O1···O3*B*	0.82	1.77	2.584 (2)	176
	O11—H11O···O3*A*	0.82	1.73	2.543 (2)	173
	N1*A*—H1N*A*···O3*A*^{ii}	0.86	2.13	2.968 (2)	163
	N1*B*—H1N*B*···O3*B*^{iii}	0.86	1.97	2.828 (2)	173
(I–*d*)	O2—H1O2···O3	0.82	1.81	2.612 (4)	166
	N1—H1N1···O3^{i}	0.86	1.91	2.767 (4)	178
(II–*b*)	O1—H1O1···O2	0.82	1.88	2.695 (2)	174
(II–*c*)	O1—H1O1···O2	0.82	1.95	2.765 (4)	173
	N1—H1N1···O2^{iv}	0.86	1.91	2.763 (2)	172
(III–*d*)	O1—H1O1···O5	0.82	1.87	2.692 (3)	178
	O2—H1O2···O1*W*^{v}	0.82	1.83	2.633 (3)	167
	O3—H1O3···O2^{vi}	0.82	2.04	2.843 (2)	167
	N1—H1N1···O5^{vii}	0.86	2.00	2.837 (3)	165
	N2—H2N2···O4^{vii}	0.86	1.97	2.829 (3)	176
	N3—H3N3···O6*A*^{viii}	0.86	1.94	2.792 (5)	174
	O1*W*—H1*W*···O1	0.93 (3)	1.89 (3)	2.817 (3)	176 (3)
	O1*W*—H2*W*···O4	0.79 (3)	2.00 (3)	2.748 (3)	159 (3)

Symmetry codes: (i) $-x+\frac{1}{2}, -y+\frac{3}{2}, -z+1$; (ii) $-x+2, -y, -z+1$; (iii) $-x+2, -y+1, -z+1$; (iv) $-x+1, -y, -z+1$; (v) $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$; (vi) $x-1, y, z$; (vii) $-x+1, -y+2, -z+1$; (viii) $-x+1, -y+1, -z+1$.

Table 2
Relevant geometric data (Å) between monomers potentially to be photodimerized.

Compound	Symmetry between molecules	Distance between reactive centers	Perpendicular distance	Lateral shift between orbitals
Ideal	**Inversion**	**3.5–4.2**	**3.5–4.2**	**0.0**
(I–*a*)	inversion	4.196 (3)	3.537 (3)	2.257 (3)
(I–*c*)	Inversion	4.865 (4)	3.552 (4)	3.324 (4)
(I–*d*)	Inversion	4.007 (6)	3.512 (6)	1.929 (6)
(II–*b*)	Inversion	4.769 (4)	3.605 (4)	3.122 (4)
(II–*c*)	Inversion	4.177 (5)	3.635 (5)	1.996 (5)
(III–*d*)[a]	None	4.027 (4)	3.634 (4)	1.735 (4)
(III–*d*)[a]	None	3.909 (4)	3.507 (4)	1.727 (4)

Note: (*a*) the two distances are not equivalent because of the absence of an inversion center.

Data collection

Nonius KappaCCD diffractometer
8509 measured reflections
3215 independent reflections
1947 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.131$
$S = 0.98$
3215 reflections
220 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

Compound (III–*d*)

Crystal data

$C_{20}H_{18}O_3 \cdot 3C_6H_7NO \cdot H_2O$
$M_r = 651.74$
Monoclinic, $P2_1/c$
$a = 10.634$ (2) Å
$b = 11.484$ (2) Å
$c = 28.574$ (4) Å
$\beta = 96.43$ (2)°
$V = 3467.5$ (10) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 293$ K
0.35 × 0.30 × 0.09 mm

Data collection

Nonius KappaCCD diffractometer
23701 measured reflections
6109 independent reflections
3757 reflections with $I > 2\sigma(I)$
$R_{int} = 0.062$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.161$
$S = 0.83$
6109 reflections
481 parameters
20 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.23$ e Å^{-3}

In (III-*d*), the occupancy factors for the disordered guest molecule were refined freely before being fixed at 76:24. The minor portion was refined with bond-length and bond-angle restraints. The H-atom positions of the major portion were calculated and fixed during the refinement. The H atoms of the minor portion were not included. The H atoms of the water molecule in (III–*d*) were refined freely. In the other compounds, all H atoms were refined at idealized positions, riding on the C, N and O atoms, with C—H distances of 0.93 and 0.96 Å, N—H distances of 0.86 Å, and O—H distances of 0.82 Å, and with U_{iso}(H) values either refined freely or set at 1.2 or 1.5 times U_{eq}(C,N,O).

For all compounds, data collection: *COLLECT* (Nonius, 2000); cell refinement: *DENZO HKL-2000* (Otwinowski & Minor, 1997); data reduction: *DENZO HKL-2000*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1999); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FG3097). Services for accessing these data are described at the back of the journal.

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