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Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.006$ Å
 R factor = 0.078
 wR factor = 0.188
Data-to-parameter ratio = 16.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

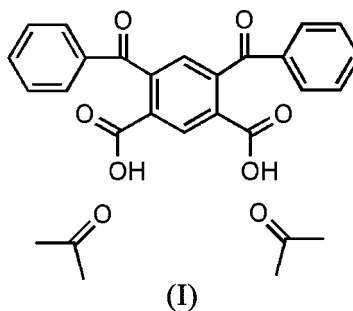
4,6-Dibenzoylisophthalic acid acetone disolvate

In the crystal structure of the title compound, $\text{C}_{22}\text{H}_{14}\text{O}_6 \cdot 2\text{C}_3\text{H}_6\text{O}$, 4,6-dibenzoylisophthalic acid is linked with two acetone solvent molecules by strong intermolecular $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds. Weak intermolecular $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds may be effective in stabilizing the crystal structure.

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Comment

4,6-Dibenzoylisophthalic acid (DBIA) and its isomer 2,5-dibenzoylterephthalic acid (DBTA) can be utilized to synthesize organic semiconductors and conjugated polymers (Tonzola *et al.*, 2003); these are of wide current interest for applications in electronic and opto-electronic devices including light-emitting diodes (Kolosov *et al.*, 2002), thin film transistors, and photovoltaic cells (Antoniadis *et al.*, 1994). We report here the crystal structure of the title compound, (I).



The molecular structure of (I) is shown in Fig. 1 and selected geometric parameters are given in Table 1. The bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The orientations of the benzoyl substituents with respect to the central benzene ring may be described by the torsion angles $\text{O}3-\text{C}13-\text{C}16-\text{C}17$ [$116.8(4)^\circ$] and $\text{O}8-\text{C}22-\text{C}20-\text{C}19$ [$110.9(5)^\circ$].

In the crystal structure, one DBIA and two acetone molecules are linked by two strong $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds (Fig. 2 and Table 2). The presence of acetone solvent molecules in the structure (in a 2:1 ratio) may play an important role in the packing mode since the numbers of hydrogen-bond acceptors and donors differ significantly from DBIA molecules themselves. Weak intermolecular $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds may be effective in the stabilization of the crystal structure.

Experimental

The title compound, (I), was prepared from a mixture of pyromellitic dianhydride (10.9 g, 50 mmol), powdered anhydrous aluminum

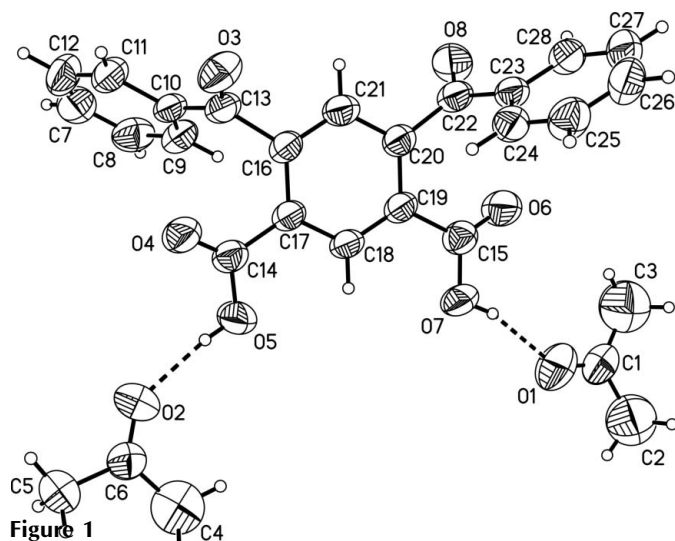


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines.

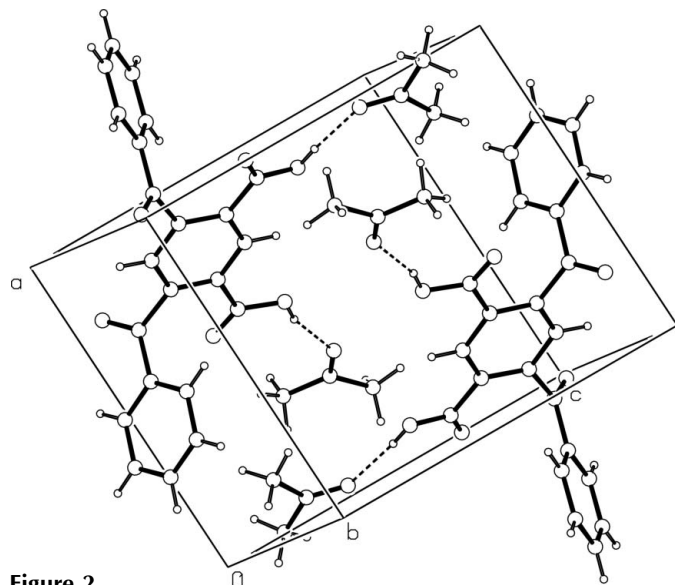


Figure 2
A packing diagram for (I). Intermolecular O—H...O hydrogen bonds are indicated by dashed lines.

chloride (30.0 g, 225 mmol) and benzene (250 ml), heated with stirring at 338–343 K for 4 h (Imai *et al.*, 1975). The reaction mixture was poured slowly into water (175 ml) containing concentrated hydrochloric acid (12.5 ml). After removal of the benzene by steam distillation, a crude product of the two acids, DBIA and DBTA, was obtained as an off-white granular material. The acids were collected by filtration, washed with water, and dissolved in dilute potassium hydroxide solution (200 ml, 5%). After filtration, the acids were reprecipitated with hydrochloric acid. The yield of the mixture of the two acids was 13.2 g (70.5%). The crude product was dissolved in methanol (87.5 ml) and a small amount of insoluble residue was removed by filtration. The solution was warmed on a water bath, and water (62.5 ml) was added. The white precipitate which formed from the clear solution upon cooling was filtered and dried. The yield of crude DBTA was 6.0 g. The filtrate was then concentrated under reduced pressure to a volume of about 75 ml. During this period, an oil separated which solidified upon cooling. The yield of the crude

acid DBIA was 5.5 g. Recrystallization of DBIA from a mixture of acetic acid/water (1:3) afforded white granules. Crystals were obtained by dissolving DBIA (1.0 g) in acetone (10 ml) and evaporating the acetone slowly at room temperature for about 30 d.

Crystal data

$C_{22}H_{14}O_6 \cdot 2C_3H_6O$
 $M_r = 490.49$
 Triclinic, $P\bar{1}$
 $a = 9.804 (2) \text{ \AA}$
 $b = 11.079 (2) \text{ \AA}$
 $c = 12.251 (3) \text{ \AA}$
 $\alpha = 102.81 (3)^\circ$
 $\beta = 93.23 (3)^\circ$
 $\gamma = 92.81 (3)^\circ$
 $V = 1292.9 (5) \text{ \AA}^3$

$Z = 2$
 $D_x = 1.260 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 10\text{--}13^\circ$
 $\mu = 0.09 \text{ mm}^{-1}$
 $T = 293.2 (1) \text{ K}$
 Block, colorless
 $0.40 \times 0.30 \times 0.20 \text{ mm}$

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.964$, $T_{\max} = 0.982$
 5363 measured reflections
 5051 independent reflections
 2541 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.063$
 $\theta_{\text{max}} = 26.0^\circ$
 $h = 0 \rightarrow 12$
 $k = -13 \rightarrow 13$
 $l = -15 \rightarrow 15$
 3 standard reflections every 200 reflections
 intensity decay: 0.1%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.078$
 $wR(F^2) = 0.188$
 $S = 1.00$
 5051 reflections
 311 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.035P)^2 + 1.9999P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.44 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.44 \text{ e \AA}^{-3}$

Table 1
Selected geometric parameters (\AA , $^\circ$).

O1—C1	1.190 (6)	O5—C14	1.316 (4)
O2—C6	1.187 (5)	O6—C15	1.193 (5)
O3—C13	1.210 (4)	O7—C15	1.318 (5)
O4—C14	1.199 (4)	O8—C22	1.214 (4)
O1—C1—C3	120.9 (5)	O4—C14—C17	123.0 (4)
O1—C1—C2	120.8 (5)	O5—C14—C17	112.6 (3)
O2—C6—C5	121.9 (5)	O6—C15—O7	123.5 (4)
O2—C6—C4	122.1 (5)	O6—C15—C19	123.6 (4)
O3—C13—C10	122.5 (3)	O7—C15—C19	112.9 (4)
O3—C13—C16	118.9 (4)	O8—C22—C23	121.9 (4)
O4—C14—O5	124.4 (4)	O8—C22—C20	119.5 (4)

Table 2
Hydrogen-bond geometry (\AA , $^\circ$).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
O5—H5D...O2	0.82	1.87	2.685 (5)	171
O7—H7B...O1	0.82	1.90	2.695 (5)	163
C9—H9A...O5 ⁱ	0.93	2.56	3.303 (5)	137
C18—H18A...O7	0.93	2.43	2.749 (5)	100
C21—H21A...O3 ⁱⁱ	0.93	2.48	3.403 (5)	172
C26—H26A...O3 ⁱⁱⁱ	0.93	2.56	3.393 (6)	149

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

H atoms were positioned geometrically, with O—H = 0.82 \AA , and C—H = 0.93 and 0.96 \AA for aromatic and methyl H atoms, respec-

tively, and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C},\text{O})$, where $x = 1.2$ for aromatic H and 1.5 for other H atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

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