2,5-Dibenzoylterephthalic acid ethanol disolvate

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The title compound, $C_{22}H_{14}O_6 \cdot 2C_2H_6O$, was synthesized by the reaction of benzene and pyromellitic acid dianhydride. The acid molecule is centrosymmetric. There are intermolecular $O-H\cdots O$ hydrogen bonds and $C-H\cdots\pi$ interactions in the crystal structure.

Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean $\sigma(C-C) = 0.004$ Å
R factor = 0.055
wR factor = 0.170
Data-to-parameter ratio = 14.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The synthesis of 2,5-dibenzoylterephthalic acid has been reported (Imai *et al.*, 1975). This paper presents the results of the single-crystal X-ray diffraction analysis of 2,5-dibenzoyl-terephthalic acid, (I).

(I)
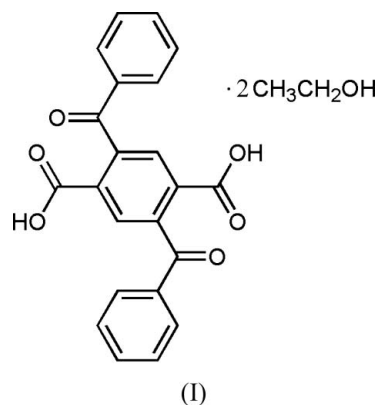
The molecular structure of (I) is shown in Fig. 1, and selected bond lengths and angles are given in Table 1. The asymmetric unit consists of one half of the molecule of (I), the molecule being centrosymmetric. An ethanol solvent molecule is also present. In the crystal structure, molecules are link by $O-H\cdots O$ and $C-H\cdots\pi$ intermolecular hydrogen bonds (Table 2), forming a two-dimensional layer structure (Fig. 2).

Experimental

A mixture of pyromellitic acid dianhydride (6.9 g, 32 mmol), powdered $AlCl_3$ (15 g, 120 mmol) and benzene (100 ml) was heated with stirring at 338–343 K for 4 h, and was then poured into water (150 ml) containing concentrated hydrochloric acid (14:1). After removing the benzene by steam distillation, the crude mixture was dissolved in dilute potassium hydroxide solution. After filtration to remove a small amount of insoluble matter, the acids were reprecipitated with hydrochloric acid. The precipitate which formed was filtered off and dried under reduced pressure. The crude product was then crystallized twice from boiling glacial acetic acid to give (I) (yield 70%). Crystals of (I) suitable for diffraction measurements were grown by slow evaporation of a solution in ethanol at room temperature.

Crystal data

$C_{22}H_{14}O_6 \cdot 2C_2H_6O$
$M_r = 466.47$
Triclinic, $P\bar{1}$
$a = 6.101\ (1)$ Å
$b = 8.598\ (2)$ Å
$c = 11.479\ (2)$ Å
$\alpha = 94.88\ (3)°$
$\beta = 98.56\ (3)°$
$\gamma = 92.42\ (3)°$
$V = 592.4\ (2)$ Å^3

$Z = 1$
$D_x = 1.308$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25
    reflections
$\theta = 10$–$13°$
$\mu = 0.10$ mm^{-1}
$T = 293\ (2)$ K
Block, colorless
$0.3 \times 0.2 \times 0.2$ mm

Data collection

Enraf–Nonius CAD-4
    diffractometer
$\omega/2\theta$ scans
Absorption correction: multi-scan
    (SADABS; Sheldrick, 2002)
    $T_{min} = 0.977$, $T_{max} = 0.981$
2555 measured reflections
2327 independent reflections
1573 reflections with $I > 2\sigma(I)$

$R_{int} = 0.024$
$\theta_{max} = 26.1°$
$h = 0 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -13 \rightarrow 14$
3 standard reflections
    every 200 reflections
    intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.170$
$S = 1.05$
2327 reflections
162 parameters

H atoms treated by a mixture of
    independent and constrained
    refinement
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
    where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.29$ e Å^{-3}
$\Delta\rho_{min} = -0.24$ e Å^{-3}

Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: (A) $1 - x$, $-y$, $-z$.]

Table 1
Selected geometric parameters (Å, °).

O1—C4	1.302 (3)	C1—C3^i	1.394 (3)
O2—C4	1.207 (3)	C3—C1^i	1.394 (3)
O3—C5	1.209 (3)	C5—C6	1.473 (3)
O2—C4—O1	124.6 (2)	O3—C5—C6	122.6 (2)
O2—C4—C3	121.4 (2)	O3—C5—C2	118.70 (19)
O1—C4—C3	114.02 (18)	C6—C5—C2	118.58 (19)

Symmetry code: (i) $-x + 1$, $-y$, $-z$.

Table 2
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
O1—H1$\cdots$O4	1.08 (4)	1.51 (4)	2.563 (3)	165 (3)
O4—H4$\cdots$O2^i	1.01 (4)	1.80 (4)	2.777 (3)	161 (4)

Symmetry code: (ii) $-x + 1$, $-y$, $-z + 1$.

H atoms attached to O atoms were located in a difference Fourier map and refined, with U_{iso}(H) values of $1.5U_{eq}$(O). H atoms bonded to C atoms were placed in calculated positions (C—H = 0.93–0.97 Å) and refined as riding, with U_{iso}(H) values of $1.2U_{eq}$(C).

Data collection: *CAD-4 Software* (Enraf–Nonius,1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms, 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*.
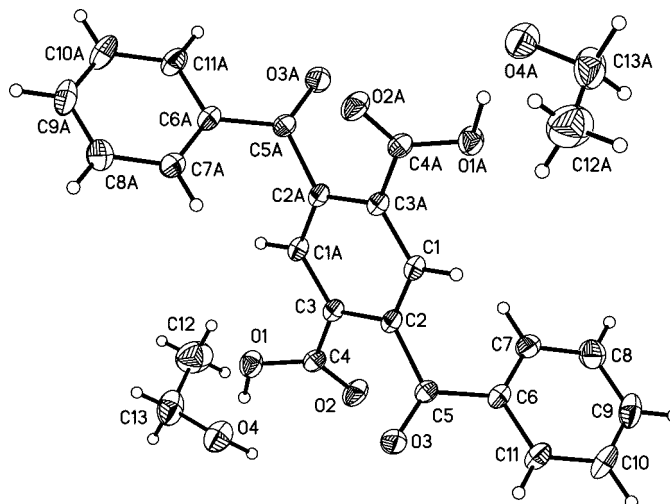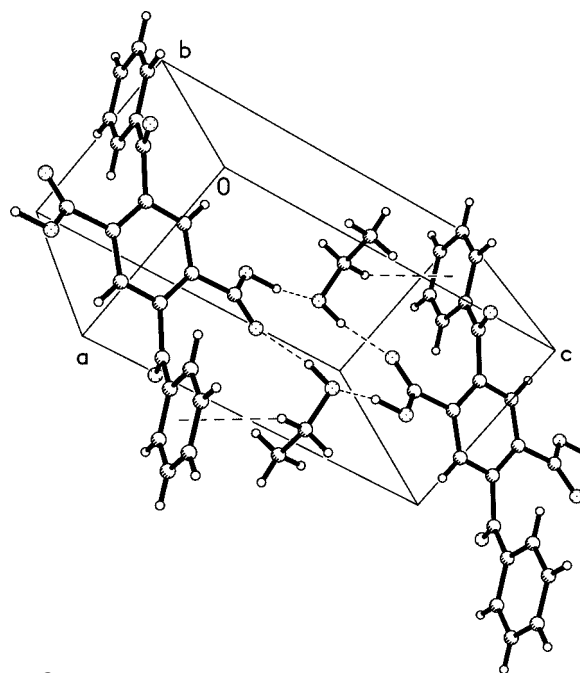

Figure 2
The crystal structure of (I). Dashed lines indicate O—H$\cdots$O hydrogen bonds and C—H$\cdots\pi$ interactions.

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