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2,5-Dibenzoylterephthalic acid dihydrate

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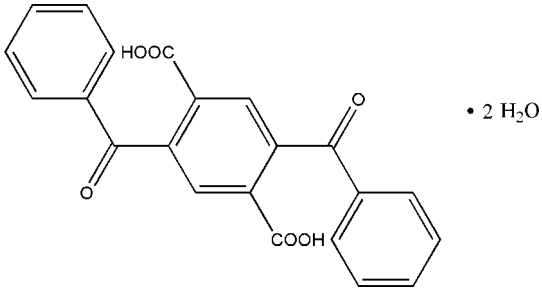
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.061; wR factor = 0.200; data-to-parameter ratio = 12.8.

The asymmetric unit of the title compound, $C_{22}H_{14}O_6\cdot 2H_2O$, contains one-half of a centrosymmetric 2,5-dibenzoylterephthalic acid molecule and one water molecule, held together by intramolecular O—H···O hydrogen bonds. The dihedral angle between the central and outer rings is 108.8 (2)°. In the crystal structure, intermolecular O—H···O hydrogen bonds link the molecules to form a three-dimensional network.

Related literature

For general background, see: Tonzola *et al.* (2003); Kolosov *et al.* (2002); Antoniadis *et al.* (1994). For related literature, see: Liu, Zhu *et al.* (2006); Liu, Heng *et al.* (2006); Liu, Ji *et al.* (2006). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{22}H_{14}O_6\cdot 2H_2O$
M_r = 410.36
Triclinic, $P\overline{1}$
a = 5.7220 (11) Å
b = 8.0630 (16) Å
c = 10.963 (2) Å
α = 102.74 (3)°
β = 101.59 (3)°
γ = 97.49 (3)°
V = 475.14 (19) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
0.40 × 0.30 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.957, T_{max} = 0.968
2056 measured reflections
1857 independent reflections
1344 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.200
S = 1.01
1857 reflections
145 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3B···OW^{i}	0.97 (5)	1.62 (5)	2.589 (4)	177 (5)
OW—HWB···O2^{ii}	0.866 (19)	1.98 (2)	2.842 (5)	171 (5)
OW—HWB···O2^{iii}	0.866 (19)	2.94 (5)	3.356 (5)	111 (4)
OW—HWA···O1	0.848 (19)	2.08 (3)	2.875 (4)	156 (5)

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

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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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2303).

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supplementary materials

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2,5-Dibenzoylterephthalic acid dihydrate

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Comment

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

The asymmetric unit of the title compound, (I), contains one half of the 2,5-dibenzoylterephthalic acid molecule and one water molecule (Fig. 1), in which they are held together by intramolecular O—H···O hydrogen bonds (Table 1). The bond lengths and angles are generally within normal ranges (Allen *et al.*, 1987). The rings A(C1—C6)and B(C8—C10/C8A—C10A) are, of course, planar and the dihedral angle between them is 108.8 (2)°, which is different significantly from the corresponding dihedral angles of 85.1 (1)° in DBTA acetic acid disolvate (Liu, Zhu *et al.*, 2006), and 83.85 (3)° in DBTA pyridine tetrasolvate (Liu, Heng *et al.*, 2006), probably due to the intramolecular O—H···O hydrogen bonds in (I).

In the crystal structure, intermolecular O—H···O hydrogen bonds (Table 1) link the molecules to form a three dimensional network (Fig. 2), in which they seem to be effective in the stabilization of the structure.

Experimental

The title compound, (I), was prepared by the literature method (Liu, Ji *et al.*, 2006). The crystals were obtained by dissolving DBTA (1.5 g, 4.0 mmol) in acetone (50 ml) with a few drops of water and evaporating the solvent slowly at room temperature for about 15 d.

Refinement

H atoms (for OH and water) were located in difference syntheses and their positions were refined [O—H = 0.848 (19)–0.97 (5) Å and U_{iso}(H) = xU_{eq}(O), where x = 1.2 for water H and x = 1.5 for OH H atoms]. The remaining H atoms were positioned geometrically, with C—H = 0.93 Å for aromatic H, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

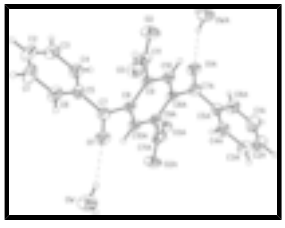


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines [symmetry code A: −*x*, −*y*, −*z*].

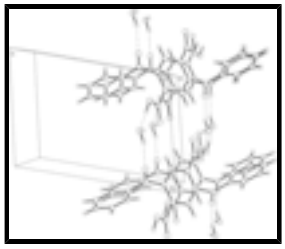


Fig. 2. A partial packing diagram of (I). Hydrogen bonds are shown as dashed lines.

2,5-Dibenzoylterephthalic acid dihydrate

Crystal data

$C_{22}H_{14}O_6 \cdot 2H_2O$	$Z = 1$
$M_r = 410.36$	$F_{000} = 214$
Triclinic, $P\overline{1}$	$D_x = 1.434$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 5.7220$ (11) Å	Cell parameters from 25 reflections
$b = 8.0630$ (16) Å	$\theta = 9$–12º
$c = 10.963$ (2) Å	$\mu = 0.11$ mm^{-1}
$\alpha = 102.74$ (3)º	$T = 298$ (2) K
$\beta = 101.59$ (3)º	Plate, colourless
$\gamma = 97.49$ (3)º	0.40 × 0.30 × 0.30 mm
$V = 475.14$ (19) Å^3	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.049$
Radiation source: fine-focus sealed tube	$\theta_{max} = 26.0$º
Monochromator: graphite	$\theta_{min} = 2.0$º
$T = 298(2)$ K	$h = -7 \rightarrow 6$
$\omega/2\theta$ scans	$k = -9 \rightarrow 9$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = 0 \rightarrow 13$
$T_{min} = 0.957$, $T_{max} = 0.968$	3 standard reflections
2056 measured reflections	every 120 min
1857 independent reflections	intensity decay: none
1344 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.061$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.200$	$w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 1.5P]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} < 0.001$
1857 reflections	$\Delta\rho_{max} = 0.38$ e Å^{-3}
145 parameters	$\Delta\rho_{min} = -0.47$ e Å^{-3}
2 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
OW	−0.1212 (6)	−0.4871 (4)	0.1929 (3)	0.0518 (8)
HWB	−0.275 (4)	−0.505 (7)	0.158 (5)	0.062*
HWA	−0.095 (9)	−0.390 (4)	0.176 (5)	0.062*
O1	0.0978 (5)	−0.1456 (4)	0.1920 (3)	0.0471 (8)
O2	0.3663 (5)	0.4355 (3)	0.0982 (3)	0.0478 (8)
O3	0.0816 (5)	0.2472 (4)	0.1329 (3)	0.0402 (7)
H3B	0.002 (9)	0.344 (7)	0.155 (5)	0.060*
C1	0.3251 (10)	0.2233 (7)	0.5483 (4)	0.0596 (13)
H1A	0.2314	0.2332	0.6089	0.072*
C2	0.5549 (10)	0.3219 (6)	0.5782 (4)	0.0568 (12)
H2A	0.6188	0.3945	0.6602	0.068*
C3	0.6902 (9)	0.3127 (6)	0.4862 (4)	0.0540 (12)
H3A	0.8431	0.3814	0.5054	0.065*
C4	0.5962 (8)	0.2005 (5)	0.3652 (4)	0.0427 (10)
H4A	0.6880	0.1930	0.3038	0.051*
C5	0.3682 (7)	0.1001 (5)	0.3351 (3)	0.0334 (8)
C6	0.2352 (8)	0.1105 (5)	0.4289 (4)	0.0463 (11)
H6A	0.0837	0.0402	0.4105	0.056*
C7	0.2633 (7)	−0.0260 (5)	0.2083 (3)	0.0337 (8)
C8	0.3820 (6)	−0.0090 (4)	0.0984 (3)	0.0282 (8)
C9	0.3948 (7)	0.1395 (4)	0.0501 (3)	0.0309 (8)
C10	0.5138 (7)	0.1452 (5)	−0.0477 (3)	0.0327 (8)
H10A	0.5236	0.2428	−0.0798	0.039*
C11	0.2797 (7)	0.2898 (4)	0.0967 (3)	0.0317 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
OW	0.0460 (17)	0.0390 (17)	0.072 (2)	0.0078 (14)	0.0242 (16)	0.0086 (15)
O1	0.0535 (18)	0.0395 (16)	0.0422 (16)	−0.0133 (13)	0.0214 (14)	0.0014 (12)
O2	0.0554 (18)	0.0273 (14)	0.070 (2)	0.0079 (12)	0.0388 (16)	0.0092 (13)
O3	0.0394 (15)	0.0353 (15)	0.0490 (17)	0.0061 (12)	0.0228 (13)	0.0065 (12)
C1	0.073 (3)	0.071 (3)	0.037 (2)	0.019 (3)	0.027 (2)	0.000 (2)
C2	0.076 (3)	0.047 (3)	0.037 (2)	0.008 (2)	0.013 (2)	−0.0086 (19)
C3	0.058 (3)	0.041 (2)	0.050 (3)	−0.003 (2)	0.004 (2)	0.000 (2)
C4	0.044 (2)	0.044 (2)	0.037 (2)	0.0030 (18)	0.0126 (17)	0.0049 (17)
C5	0.039 (2)	0.0338 (19)	0.0284 (18)	0.0058 (15)	0.0140 (15)	0.0051 (15)
C6	0.051 (3)	0.049 (2)	0.037 (2)	−0.0005 (19)	0.0256 (19)	−0.0011 (18)
C7	0.0341 (19)	0.0334 (19)	0.0322 (19)	−0.0014 (15)	0.0145 (15)	0.0037 (15)
C8	0.0328 (18)	0.0234 (16)	0.0266 (17)	0.0016 (13)	0.0131 (14)	−0.0010 (13)
C9	0.0370 (19)	0.0253 (17)	0.0277 (18)	0.0014 (14)	0.0123 (15)	−0.0003 (14)
C10	0.038 (2)	0.0299 (18)	0.0299 (18)	0.0044 (15)	0.0144 (15)	0.0026 (14)
C11	0.038 (2)	0.0273 (18)	0.0307 (18)	0.0111 (15)	0.0130 (15)	0.0010 (14)

Geometric parameters (Å, °)

OW—HWB	0.866 (19)	C5—C4	1.378 (5)
OW—HWA	0.85 (4)	C5—C6	1.391 (5)
O1—C7	1.215 (4)	C6—C1	1.374 (6)
O2—C11	1.209 (4)	C6—H6A	0.9300
O3—C11	1.304 (4)	C7—C5	1.486 (5)
O3—H3B	0.97 (5)	C7—C8	1.520 (5)
C1—H1A	0.9300	C8—C10^{i}	1.380 (5)
C2—C1	1.381 (7)	C9—C10	1.387 (5)
C2—H2A	0.9300	C9—C8	1.412 (5)
C3—C2	1.385 (7)	C9—C11	1.494 (5)
C3—H3A	0.9300	C10—C8^{i}	1.380 (5)
C4—C3	1.387 (6)	C10—H10A	0.9300
C4—H4A	0.9300		
HWB—OW—HWA	93 (5)	C1—C6—H6A	119.6
C11—O3—H3B	113 (3)	C5—C6—H6A	119.6
C6—C1—C2	119.8 (4)	O1—C7—C5	122.7 (3)
C6—C1—H1A	120.1	O1—C7—C8	119.6 (3)
C2—C1—H1A	120.1	C5—C7—C8	117.5 (3)
C1—C2—C3	120.1 (4)	C10^{i}—C8—C9	119.8 (3)
C1—C2—H2A	120.0	C10^{i}—C8—C7	117.1 (3)
C3—C2—H2A	120.0	C9—C8—C7	123.0 (3)
C2—C3—C4	119.6 (4)	C10—C9—C8	118.8 (3)
C2—C3—H3A	120.2	C10—C9—C11	118.1 (3)
C4—C3—H3A	120.2	C8—C9—C11	123.1 (3)
C5—C4—C3	120.7 (4)	C8^{i}—C10—C9	121.4 (3)

C5—C4—H4A	119.7	C8^{i}—C10—H10A	119.3
C3—C4—H4A	119.7	C9—C10—H10A	119.3
C4—C5—C6	119.0 (4)	O2—C11—O3	124.8 (3)
C4—C5—C7	122.6 (3)	O2—C11—C9	121.4 (3)
C6—C5—C7	118.4 (3)	O3—C11—C9	113.7 (3)
C1—C6—C5	120.8 (4)		
C8—C9—C10—C8^{i}	0.2 (6)	C5—C7—C8—C9	−61.4 (5)
C11—C9—C10—C8^{i}	−178.0 (3)	O1—C7—C5—C4	159.5 (4)
C10—C9—C11—O2	−32.9 (5)	C8—C7—C5—C4	−16.3 (6)
C8—C9—C11—O2	148.9 (4)	O1—C7—C5—C6	−17.3 (6)
C10—C9—C11—O3	146.5 (3)	C8—C7—C5—C6	166.9 (4)
C8—C9—C11—O3	−31.7 (5)	C6—C5—C4—C3	−1.2 (6)
C10—C9—C8—C10^{i}	−0.2 (6)	C7—C5—C4—C3	−178.0 (4)
C11—C9—C8—C10^{i}	177.9 (3)	C4—C5—C6—C1	2.3 (7)
C10—C9—C8—C7	178.0 (3)	C7—C5—C6—C1	179.2 (4)
C11—C9—C8—C7	−3.9 (5)	C5—C4—C3—C2	1.0 (7)
O1—C7—C8—C10^{i}	−59.1 (5)	C4—C3—C2—C1	−1.8 (7)
C5—C7—C8—C10^{i}	116.8 (4)	C5—C6—C1—C2	−3.1 (8)
O1—C7—C8—C9	122.6 (4)	C3—C2—C1—C6	2.8 (8)

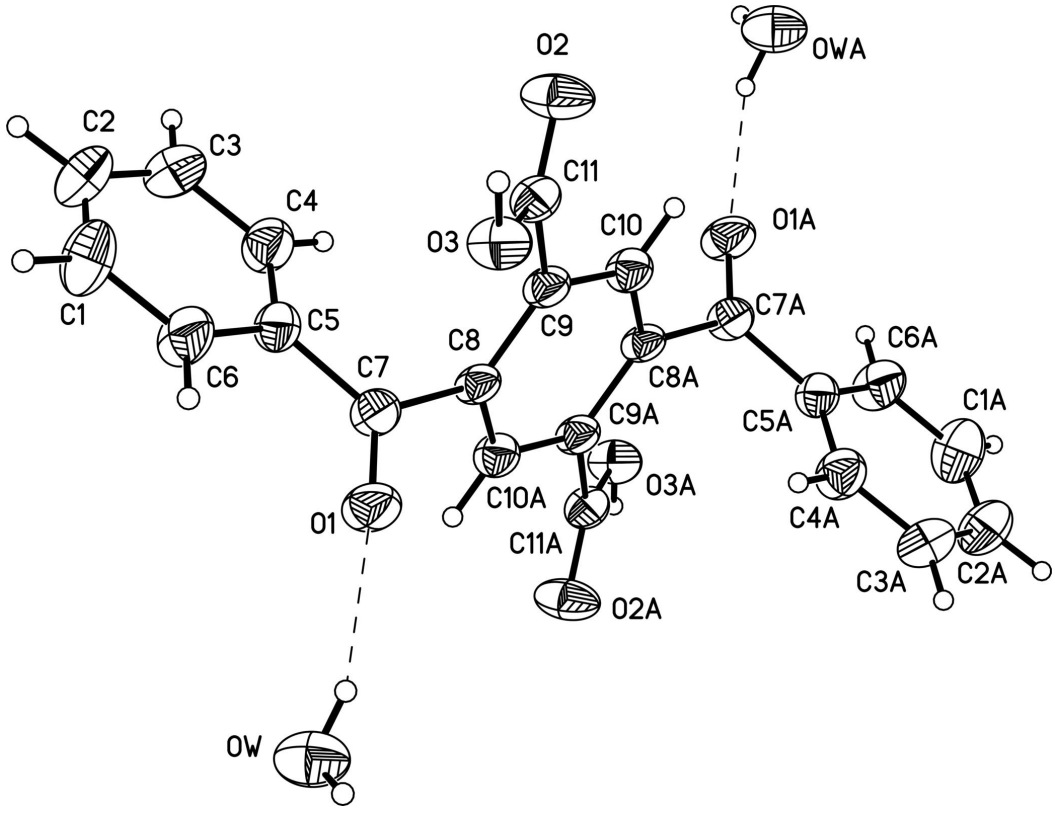
Symmetry codes: (i) −*x*+1, −*y*, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3B···OW^{ii}	0.97 (5)	1.62 (5)	2.589 (4)	177 (5)
OW—HWB···O2^{iii}	0.866 (19)	1.98 (2)	2.842 (5)	171 (5)
OW—HWB···O2^{iv}	0.866 (19)	2.94 (5)	3.356 (5)	111 (4)
OW—HWA···O1	0.848 (19)	2.08 (3)	2.875 (4)	156 (5)

Symmetry codes: (ii) *x*, *y*+1, *z*; (iii) *x*−1, *y*−1, *z*; (iv) −*x*, −*y*, −*z*.

Fig. 1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OWA
C2
C3
C11
C4
O3
C10
O1A
C1
C5
C8
C9
C7A
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C6
C7
C8A
C1A
C9A
C5A
O3A
C10A
C4A
O1
C11A
C3A
C2A
O2A
OW

Fig. 2