

Shan Liu, Guang-Liang Song,
Jun-Mei Tang, Wen-Kai Xu and
Hong-Jun Zhu*Department of Applied Chemistry, College of
Science, Nanjing University of Technology,
Nanjing 210009, People's Republic of China

Correspondence e-mail: zhuhj@njut.edu.cn

Key indicators

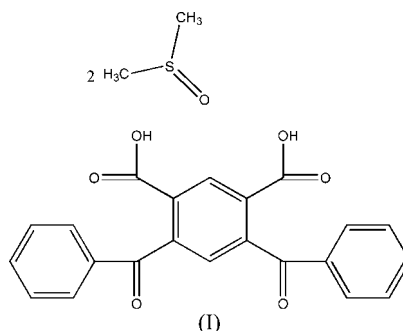
Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
Disorder in solvent or counterion
 R factor = 0.103
 wR factor = 0.217
Data-to-parameter ratio = 17.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.4,6-Dibenzoylisophthalic acid dimethyl
sulfoxide disolvate

In the crystal structure of the title compound, $\text{C}_{22}\text{H}_{14}\text{O}_6 \cdot 2 \cdot 2\text{C}_2\text{H}_6\text{OS}$, 4,6-dibenzoylisophthalic acid is connected to two dimethyl sulfoxide molecules by strong $\text{O}-\text{H} \cdots \text{O}=\text{S}$ hydrogen bonds. Weak intermolecular $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds connect molecules into dimers. Intramolecular three-centred $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonding is favoured by a *syn* conformation of both carboxyl groups. The two dimethyl sulfoxide molecules were found to be disordered over two equally populated sites.

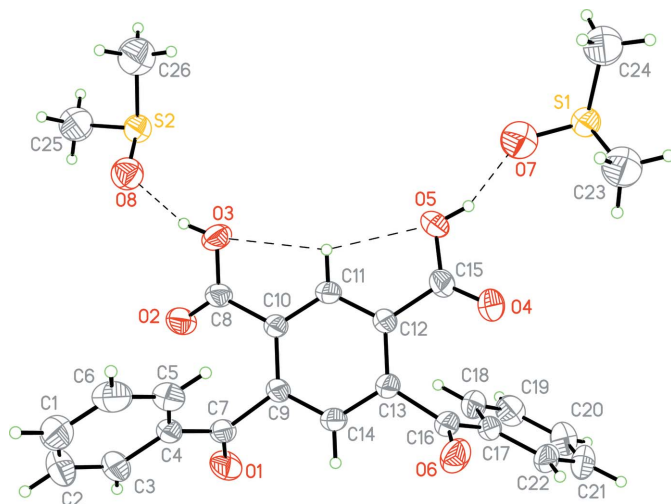
Received 29 April 2006
Accepted 30 May 2006

Comment

4,6-Dibenzoylisophthalic acid (DBIA) and its isomer 2,5-dibenzoyltetraphthalic acid (DBTA) can be utilized to synthesize organic semiconductors and conjugated polymers (Christopher *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 here the crystal structure of the title compound, (I) (Fig. 1). The bond lengths and angles are within normal ranges (Allen *et al.*, 1987). The orientations of the benzoyl substituents with respect to the benzene rings are described by the torsion angles $\text{C}12-\text{C}13-\text{C}16-\text{O}6$ [109.6 (5°)] and $\text{O}1-\text{C}7-\text{C}9-\text{C}10$ [121.4 (5°)]. In the crystal structure, one DBIA and two dimethyl sulfoxide molecules are linked by two strong $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds (Fig. 1 and Table 1). The presence of dimethyl sulfoxide molecules in a 2:1 molar ratio and their disorder could be related to the number of available donor and acceptor sites to satisfy hydrogen bonding. The intramolecular bifurcated hydrogen bond (Fig. 1), $\text{C}11-\text{H}11\text{A} \cdots \text{O}5$ and $\text{C}11-\text{H}11\text{A} \cdots \text{O}3$, stabilizes the molecular conformation, whereas intermolecular interaction $\text{C}14-\text{H}14\text{A} \cdots \text{O}1^i$ creates a dimer (Fig. 2 and Table 1). The intermolecular and intramolecular $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds (Table 1) influence the crystal packing of (I) more than the weak intermolecular $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds between DBIA and two acetone molecules that we reported recently (Liu *et al.*, 2006).


Figure 1

A view of (I), with the atom numbering scheme and one of two possible positions of the solvent molecules shown. Displacement ellipsoids are drawn at the 40% probability level. Hydrogen bonds are shown by dashed lines.

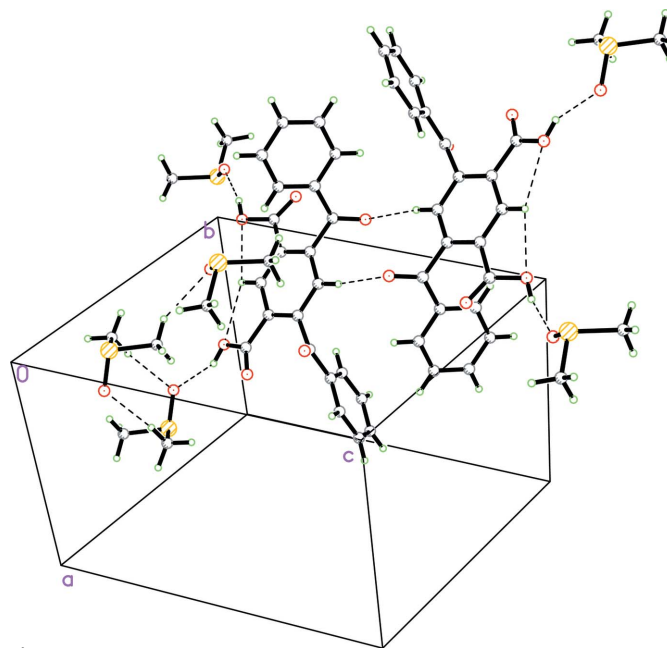
Experimental

Compound (I) was prepared from a mixture of pyromellitic dianhydride (10.9 g, 50 mmol), powdered anhydrous aluminium 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 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, 5.0%). 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 that formed in the clear solution upon cooling was filtered off 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 dimethyl sulfoxide (25 ml) and evaporating the solvent at room temperature for about 60 d.

Crystal data

$C_{22}H_{14}O_6 \cdot 2C_2H_6OS$
 $M_r = 530.59$
 Triclinic, $P\bar{1}$
 $a = 9.880$ (2) Å
 $b = 10.858$ (2) Å
 $c = 12.627$ (3) Å
 $\alpha = 75.45$ (3)°
 $\beta = 89.59$ (3)°
 $\gamma = 84.74$ (3)°

$V = 1305.4$ (5) Å³
 $Z = 2$
 $D_x = 1.350$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.25$ mm⁻¹
 $T = 293$ (2) K
 Block, colourless
 0.30 × 0.20 × 0.10 mm


Figure 2

The packing of (I), showing O—H...O intermolecular hydrogen bonds and C—H...O hydrogen bonds (dashed lines).

Data collection

Enraf–Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{\min} = 0.929$, $T_{\max} = 0.975$
 5112 measured reflections

5112 independent reflections
 3100 reflections with $I > 2\sigma(I)$
 $\theta_{\max} = 26.0^\circ$
 3 standard reflections
 every 200 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.103$
 $wR(F^2) = 0.217$
 $S = 1.02$
 5112 reflections
 301 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + 5P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.006$
 $\Delta\rho_{\max} = 0.82$ e Å⁻³
 $\Delta\rho_{\min} = -0.92$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H3B...O8	0.82	1.80	2.594 (8)	162
O5—H5B...O7	0.82	1.83	2.584 (9)	153
C11—H11A...O5	0.93	2.42	2.740 (6)	100
C11—H11A...O3	0.93	2.44	2.751 (6)	99
C14—H14A...O1 ⁱ	0.93	2.52	3.442 (5)	171
C23—H23B...O7 ⁱⁱ	0.96	2.54	3.497 (13)	174
C24—H24B...O8 ⁱⁱⁱ	0.96	2.59	3.368 (14)	138

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

The two dimethyl sulfoxide molecules were found to be disordered over two equally populated positions: C23/C23', C24/C24', C25/C25', C26/C26', S1/S1', S2/S2', O7/O7' and O8/O8'. The C—S and O—S bond lengths involving the disordered atoms were restrained to 1.50 (6) Å and 1.39 (7) Å, respectively. The U^{ij} components of the disordered atoms were approximated to isotropic behaviour. H atoms were placed at idealized positions and allowed to ride on their parent

atoms, with C—H = 0.93 and 0.96 Å, O—H = 0.82 Å and $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{C},\text{O})$.

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*.

The authors thank the Center of Testing and Analysis, Nanjing University, for the support.

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
- Antoniadis, H., Hsieh, B. R., Abkowitz, M. A., Jenekhe, S. A. & Stolka, M. (1994). *Synth. Met.* **62**, 265–271.
- Bruker (2000). *XSCANS* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Christopher, J. T., Maksudul, M. A., Werner, K. & Samson, A. J. (2003). *J. Am. Chem. Soc.* **125**, 13548–13558.
- Enraf–Nonius (1985). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
- Imai, Y., Johnson, E. F., Katto, T., Kurihara, M. & Stille, J. K. (1975). *J. Polym. Sci. Part A Polym. Chem.* **13**, 2233–2249.
- Kolosov, S., Adamovich, V., Djurovich, P., Thompson, M. E. & Adachi, C. (2002). *J. Am. Chem. Soc.* **124**, 9945–9954.
- Liu, S., Ji, J. X., Wang, D. D. & Zhu, H. J. (2006). *Acta Cryst.* **E62**, o1387–o1389.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.