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N,*N*′-Bis(3-hydroxyphenyl)-1,8:4,5-naphthalenetetracarboximide dimethyl sulfoxide disolvate

Gary D. Fallon,[a]* Marcia A.-P. Lee[b] and Steven J. Langford[a]

[a]School of Chemistry, Building 23, Monash University, Victoria 3800, Australia, and [b]Centre for Green Chemistry, Building 23, Monash University, Victoria 3800, Australia

Correspondence e-mail: g.fallon@sci.monash.edu.au

Key indicators

Single-crystal X-ray study
T = 123 K
Mean σ(C–C) = 0.002 Å
R factor = 0.039
wR factor = 0.105
Data-to-parameter ratio = 17.5

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

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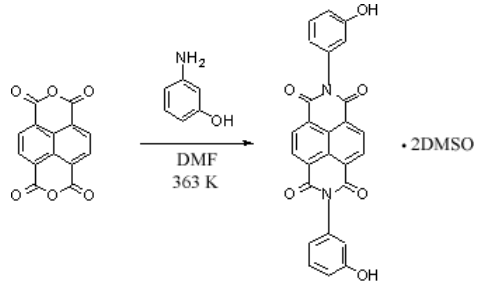
The structure of the title compound, $C_{26}H_{14}N_2O_6\cdot 2C_2H_6OS$, shows the essentially planar molecule to be centrosymmetric and hydrogen-bonded *via* the phenolic H atom to DMSO molecules.

Comment

Aromatic diimides such as the naphthalene diimides (NDI) are neutral, planar, redox-active molecules whose structural and electronic properties have had implications in areas as diverse as chemotherapeutic agents and photosynthetic mimics (Vicic *et al.*, 2000; Osuka *et al.*, 1995; Hamilton *et al.*, 1998). From our perspective, we were interested in preparing a range of copolymers based on the diphenol (I), onto which wheel components, such as crown ethers, could be threaded. The polypseudorotaxanes formed in this way may have interesting physical properties which differ from those of the 'axle' component. The synthesis of (I) was efficiently achieved by condensing commercially available 1,4,5,8-naphthalenetetracarboxylic dianhydride with 3-aminophenol in hot DMF solvent. The low solubility of the product in water allows for easy isolation of (I) from the DMF solvent. Vapour diffusion of H_2O into a DMSO solution of (I) afforded single crystals suitable for X-ray analysis.



(I)

The centrosymmetric molecular structure of (I) is shown in Fig. 1. Clearly indicated along with (I) are DMSO solvent molecules of crystallization, which are connected through a hydrogen bonding interaction between the phenolic H atoms of (I) and the DMSO oxygen (H···O4 = 1.78 Å, S—O···O = 2.6186 (17) Å and the angle subtended at H = 177°). The supramolecular structure of (I)·2DMSO shows that there is very little interaction between neighbouring diimides within the crystal lattice. The central core of the naphthalene diimide is essentially planar, the maximum deviation being 0.09 (2) Å for C6. The carboxylic O atoms are twisted out of the plane by −0.25 (1) and 0.19 (1) Å. The dihedral angle between the diimide plane and that of the benzene ring is 62.4 (1)°.

Experimental

3–Aminophenol (0.50 g, 4.58 mmol) was added to a stirred solution of 1,4,5,8-naphthalenetetracarboxylic dianhydride (0.52 g, 1.94 mmol) in dry DMF (20 ml). The reaction mixture was then warmed to 363 K (complete dissolution) and stirred overnight. Once the reaction was complete, water (20 ml) was added and the reaction mixture allowed to cool to room temperature. The resulting precipitate was collected at the pump, washed with water and dried to yield (I) (0.70 g, 80%).

Crystal data

$C_{26}H_{14}N_2O_6\cdot 2C_2H_6OS$	D_x = 1.477 Mg m^{-3}
M_r = 606.65	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 19123 reflections
a = 14.1659 (2) Å	θ = 2.9–28.3°
b = 7.9501 (1) Å	μ = 0.25 mm^{-1}
c = 12.3307 (2) Å	T = 123 (2) K
β = 100.850 (1)°	Needle, yellow-brown
V = 1363.86 (3) Å^3	0.28 × 0.10 × 0.07 mm
Z = 2	

Data collection

Nonius KappaCCD diffractometer	R_{int} = 0.054
Thick-slice φ and ω scans	θ_{max} = 28°
19 123 measured reflections	$h = -18 \rightarrow 18$
3375 independent reflections	$k = -10 \rightarrow 10$
2478 reflections with $I > 2\sigma(I)$	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 0.1051P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.039	$(\Delta/\sigma)_{max}$ = 0.002
$wR(F^2)$ = 0.105	$\Delta\rho_{max}$ = 0.40 e Å^{-3}
S = 1.09	$\Delta\rho_{min}$ = −0.40 e Å^{-3}
3375 reflections	
193 parameters	
H-atom parameters constrained	

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3···O4	0.84	1.78	2.6186 (17)	177

H atoms were included in the riding-model approximation.

Data collection: *COLLECT* (Nonius, 1997–2002); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

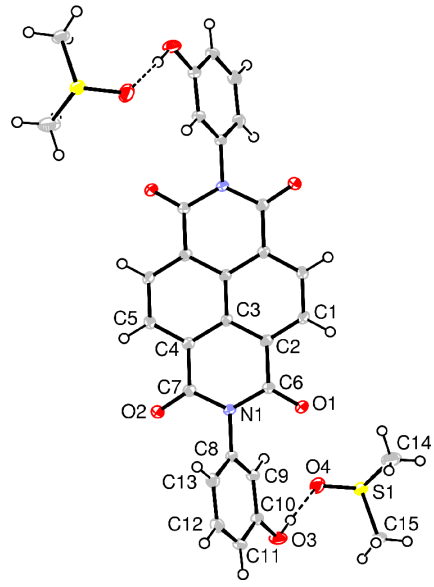


Figure 1
View of (I)·2DMSO, with 50% probability displacement ellipsoids.

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