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A 1:2 complex of pyromellitic diimide and dimethyl sulfoxide

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Key indicators

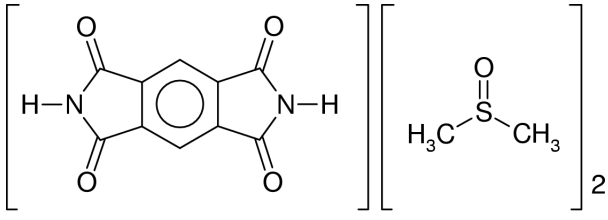
Single-crystal X-ray study
T = 180 K
Mean σ(C–C) = 0.003 Å
R factor = 0.045
wR factor = 0.108
Data-to-parameter ratio = 16.3

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

Recrystallization of pyromellitic diimide from dimethyl sulfoxide yields a 1:2 complex, $C_{10}H_4N_2O_4{\cdot}2C_2H_6SO$. In space group $P2_1/c$, the diimide molecules form herring-bone-packed layers and are capped at each end by hydrogen-bonded dimethyl sulfoxide molecules. The 1:2 complex is sited on a crystallographic centre of symmetry.

Comment

Recrystallization of pyromellitic diimide (PMDI), $C_{10}H_4N_2O_4$, from dimethyl sulfoxide (DMSO) solution, C_2H_6SO, yields a 1:2 complex of the two components (Fig. 1). The N—H groups at both ends of each PMDI molecule are involved in hydrogen bonds to O atoms of DMSO molecules [H1N···O3 = 1.98 (2) Å and N1—H1N···O3 = 174 (2)°]. One methyl group of each DMSO molecule is also directed towards one C=O group of the diimide, suggestive of a weak hydrogen-bond interaction. The 1:2 complex is sited on a crystallographic centre of symmetry in space group $P2_1/c$.



(I)

The structure, (I), may be envisaged as layers of PMDI molecules packed in a herring-bone arrangement parallel to (100) (Fig. 2), with DMSO molecules sited between the layers (Fig. 3). Alternatively, the 1:2 complexes may be viewed as discrete supermolecules that form herring-bone-packed layers stacked along the a axis.

Experimental

Crystals were grown by slow evaporation at room temperature of a solution of pyromellitic diimide (obtained from the Aldrich Co.) in anhydrous dimethyl sulfoxide.

$C_{10}H_4N_2O_4{\cdot}2C_2H_6OS$
M_r = 372.41
Monoclinic, $P2_1/c$
a = 12.4152 (11) Å
b = 5.3266 (2) Å
c = 13.8046 (12) Å
β = 114.533 (3)°
V = 830.49 (11) Å^3
Z = 2
D_x = 1.489 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2650 reflections
θ = 1.0–27.5°
μ = 0.35 mm^{-1}
T = 180 (2) K

Crystal data

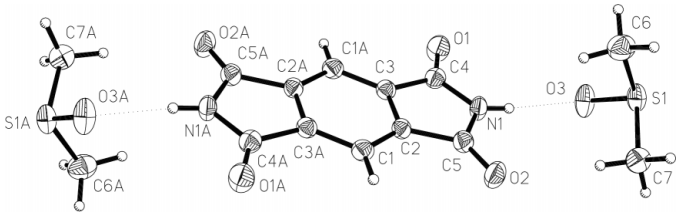


Figure 1
The molecular structure of the 1:2 complex showing displacement ellipsoids at the 50% probability level for non-H atoms (*XP*; Sheldrick, 1993). Atoms related by the centre of symmetry are denoted by the suffix A.

Block, colourless	0.23 × 0.23 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer	1302 reflections with $I > 2\sigma(I)$
Thin-slice ω and φ scans	$R_{int} = 0.045$
Absorption correction: multi-scan (*SORTAV*; Blessing, 1995)	$\theta_{max} = 27.5°$
$T_{min} = 0.879$, $T_{max} = 0.958$	$h = -16 \rightarrow 15$
5086 measured reflections	$k = -5 \rightarrow 6$
1886 independent reflections	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.045$	$(\Delta/\sigma)_{max} = 0.010$
$wR(F^2) = 0.108$	$\Delta\rho_{max} = 0.27$ e Å^{-3}
$S = 1.12$	$\Delta\rho_{min} = -0.39$ e Å^{-3}
1886 reflections	Extinction correction: *SHELXL*97
116 parameters	Extinction coefficient: 0.038 (4)
H atoms treated by a mixture of independent and constrained refinement	

The H atom associated with the imide group (H1N) was located from a difference Fourier map and refined freely with an isotropic displacement parameter. All H atoms attached to C were placed geometrically and refined using a riding model with U_{iso}(H) = xU_{eq}(C), where x = 1.2 for H1*A* and 1.5 for methyl H atoms. C—H distances were fixed at 0.95 Å for H1*A* and 0.98 Å for methyl H atoms, and the methyl groups were allowed to rotate about their local threefold axes.

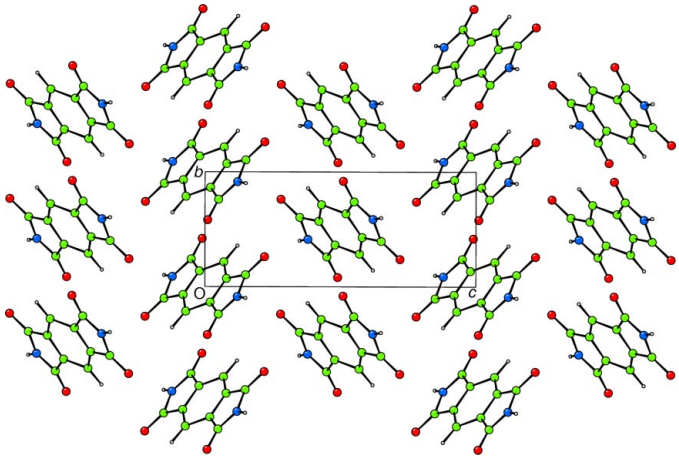


Figure 2
Projection on to (100) of a single layer of PMDI molecules (DMSO molecules omitted) showing the herring-bone arrangement (*CAMERON*; Watkin *et al.*, 1996).

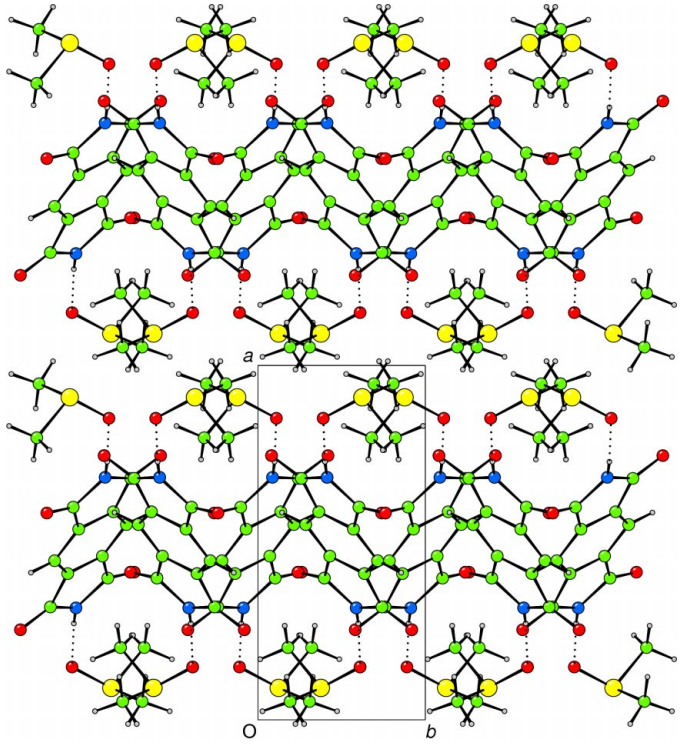


Figure 3
Projection on to (001) showing alternate layers of PMDI and DMSO molecules stacked along the *a* axis (*CAMERON*; Watkin *et al.*, 1996). Key: green: C; blue: N; red: O; yellow: S; grey: H.

Data collection: *COLLECT* (Nonius, 1998); 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: XP (Sheldrick, 1993) and *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *SHELXL*97.

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