Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

N,*N*′-Bis(2-pyridylmethyl)pyromellitic diimide

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Received 28 October 2003
Accepted 29 October 2003
Online 8 November 2003

Key indicators

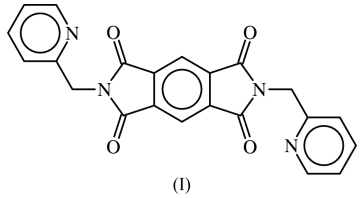
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.004 Å
R factor = 0.033
wR factor = 0.091
Data-to-parameter ratio = 7.6

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

The title compound, $C_{22}H_{14}N_4O_4$, exists as a monomeric molecule in which the pyridyl rings are approximately perpendicular to the pyromellitic diimide portion [dihedral angles 83.7 (1) and 85.3 (1)°]. Adjacent molecules are linked by two C—H···O interactions into a linear chain parallel to the *b* axis.

Comment

Pyromellitic diimide (Borkent *et al.*, 1976) can be easily di-alkylated to give compounds that are electron acceptors, a property that can be exploited for the synthesis of electron-donor/spacer/electron-acceptor compounds (Hunter *et al.*, 1989). The crystal structure of the ethyl homolog is known from the crystal structure of its adduct with 2,6-dimethoxy-naphthalene (Hamilton *et al.*, 1998). The self-assembly of *N*,*N*′-bis(2-*tert*-butylphenyl)pyromellitic diimide, phenols and indoles is another example that displays host–guest interactions leading to a stacked sandwich structure (Kishikawa *et al.*, 1999). Our purpose in using the pyridyl-2-aminomethyl group as the substituent is to synthesize a bis-pyridyl compound in which the two heterocyclic entities are separated by a rigid aromatic system, with the requirement that the rings are capable of free rotation. The expected compound, (I) (see scheme), exists in the solid state as a monomeric molecule. The pyromelletic diimide portion is planar, and both of the pyridyl rings are approximately perpendicular to this plane [dihedral angles 83.7 (1) and 85.3 (1)°], but one is above and the other is below the plane (Fig. 1). The molecules are linked by two C—H···O interactions to furnish a chain running along the *b* axis.



(I)

Experimental

The compound was prepared by the condensation of pyromellitic dianhydride (8.74 g, 40 mmol) and aminomethyl-2-pyridine (9.0 g, 80 mmol) in DMF (40 ml). The reaction mixture was heated for 5 h. A solid product separated on cooling and this was collected by filtration. The compound was purified by recrystallization twice, once from DMF and the second time from chloroform, to give the pure compound in 80% yield. Elemental analysis for $C_{22}H_{14}N_4O_4$, found: C 66.24, H 3.51, N 14.19%; calculated: C 66.33, H 3.52, N 14.07%.

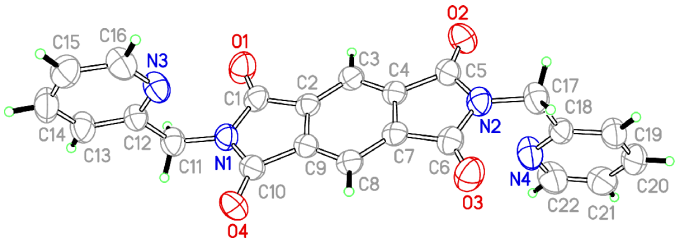


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I), with ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

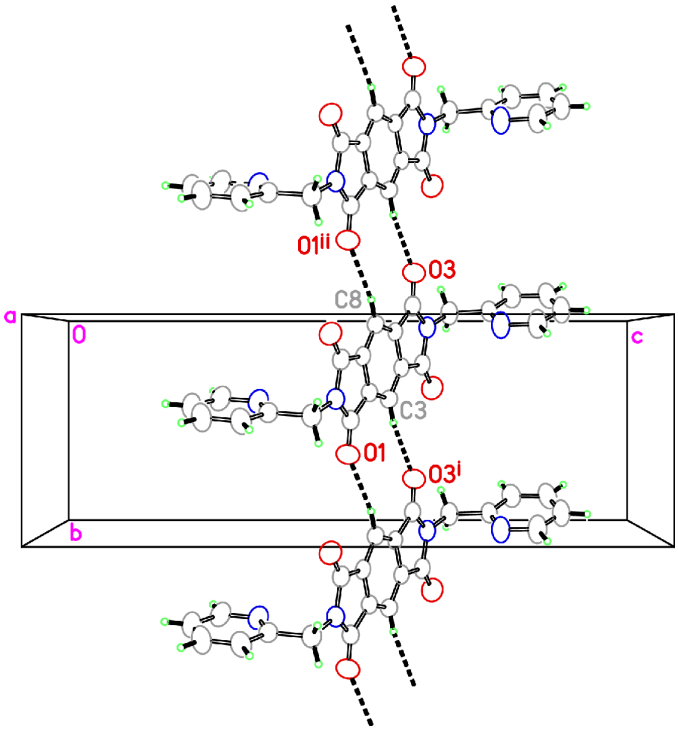


Figure 2
*ORTEP*II (Johnson, 1976) plot showing the C—H···O interactions that link the molecules into a chain running along the *b* axis; C3···O3^{i} = 3.241 (4) Å and C8···O1^{ii} = 3.253 (3) Å. [Symmetry code: (i) *x*, 1 + *y*, *z*; (ii) *x*, *y* − 1, *z*.]

Crystal data

$C_{22}H_{14}N_4O_4$
M_r = 398.37
Orthorhombic, $Pca2_1$
a = 11.749 (1) Å
b = 7.498 (2) Å
c = 21.028 (3) Å
V = 1852.3 (5) Å^3
Z = 4
D_x = 1.428 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 861 reflections
θ = 2.6–26.6°
μ = 0.10 mm^{-1}
T = 298 (2) K
Prism, colorless
0.30 × 0.24 × 0.15 mm

Data collection

Bruker SMART area-detector diffractometer
φ and ω scans
Absorption correction: none
8901 measured reflections
2062 independent reflections
1610 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 27.0°
h = −15 → 11
k = −6 → 9
l = −26 → 26

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.091
S = 1.02
2062 reflections
271 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0512P)^2 + 0.1847P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.13 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.208 (3)	C3—C4	1.386 (4)
O2—C5	1.199 (3)	C4—C7	1.390 (3)
O3—C6	1.208 (3)	C4—C5	1.493 (4)
O4—C10	1.209 (3)	C6—C7	1.480 (4)
N1—C1	1.380 (4)	C7—C8	1.387 (4)
N1—C10	1.391 (4)	C8—C9	1.378 (4)
N1—C11	1.446 (4)	C9—C10	1.491 (4)
N2—C5	1.395 (4)	C11—C12	1.510 (4)
N2—C6	1.387 (4)	C12—C13	1.378 (4)
N3—C12	1.317 (4)	C13—C14	1.370 (4)
N3—C16	1.343 (4)	C14—C15	1.369 (5)
N2—C17	1.443 (4)	C15—C16	1.363 (5)
N4—C18	1.323 (4)	C17—C18	1.518 (4)
N4—C22	1.348 (4)	C18—C19	1.365 (4)
C1—C2	1.492 (4)	C19—C20	1.373 (5)
C2—C3	1.384 (4)	C20—C21	1.363 (5)
C2—C9	1.385 (4)	C21—C22	1.357 (5)
C1—N1—C10	111.9 (2)	C4—C7—C6	107.9 (2)
C1—N1—C11	123.5 (2)	C6—C7—C8	129.7 (2)
C10—N1—C11	124.0 (2)	C7—C8—C9	114.8 (2)
C5—N2—C6	112.0 (2)	C8—C9—C2	122.9 (2)
C5—N2—C17	123.8 (2)	C8—C9—C10	129.5 (2)
C6—N2—C17	123.7 (3)	C2—C9—C10	107.5 (2)
C12—N3—C16	116.8 (2)	O4—C10—N1	125.2 (3)
C18—N4—C22	115.9 (3)	O4—C10—C9	128.5 (3)
O1—C1—N1	125.1 (3)	N1—C10—C9	106.2 (2)
O1—C1—C2	128.8 (3)	N1—C11—C12	113.8 (2)
N1—C1—C2	106.2 (2)	N3—C12—C13	123.2 (3)
C3—C2—C9	122.7 (2)	N3—C12—C11	117.5 (2)
C1—C2—C3	129.3 (2)	C11—C12—C13	119.3 (3)
C1—C2—C9	108.0 (2)	C12—C13—C14	119.0 (3)
C2—C3—C4	114.6 (2)	C13—C14—C15	118.7 (3)
C3—C4—C5	129.4 (2)	C16—C15—C14	118.6 (3)
C3—C4—C7	122.7 (3)	N3—C16—C15	123.7 (3)
C5—C4—C7	107.9 (2)	N2—C17—C18	114.1 (2)
O2—C5—N2	125.5 (3)	N4—C18—C19	123.5 (3)
O2—C5—C4	128.8 (3)	N4—C18—C17	117.6 (2)
N2—C5—C4	105.7 (2)	C17—C18—C19	118.9 (3)
O3—C6—N2	124.6 (3)	C18—C19—C20	119.3 (3)
O3—C6—C7	128.9 (3)	C19—C20—C21	118.4 (3)
N2—C6—C7	106.5 (2)	C22—C21—C22	118.7 (3)
C4—C7—C8	122.3 (2)	N4—C22—C21	124.2 (3)

The H atoms were placed at calculated positions in the riding-model approximation (C—H = 0.93 Å for the aromatic H atoms and C—H 0.97 Å for the aliphatic H atoms), and their displacement parameters were set to 1.2 times U_{eq} of their parent atoms.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 1999); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

The authors thank the National Natural Science Foundation of China, the Natural Science Foundation of Guangdong Province, Sun Yat-Sen University and the University of Malaya for supporting this work.

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