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N,*N*′-Di-4-pyridylperylene-3,4:9,10-bis-(dicarboximide)

Kazuyuki Hino, Kazuyuki Sato, Hiroo Takahashi, Shigeru Suzuki and Jin Mizuguchi*

Department of Applied Physics, Graduate School of Engineering, Yokohama National University, 79-5 Tokiwadai, Hodogaya-ku, 240-8501 Yokohama, Japan

Correspondence e-mail: mizu-j@ynu.ac.jp

Key indicators

Single-crystal X-ray study
T = 93 K
Mean σ(C–C) = 0.007 Å
R factor = 0.074
wR factor = 0.147
Data-to-parameter ratio = 10.3

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

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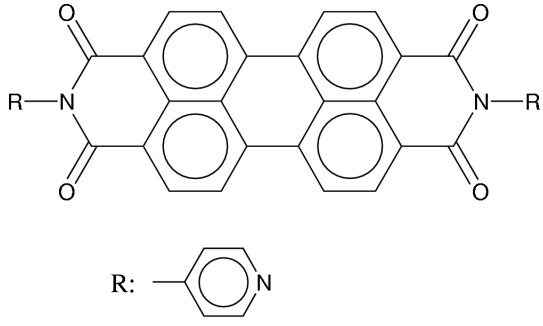
The title compound, $C_{34}H_{16}N_4O_4$, is a perylene–imide pigment utilized for H_2 gas sensors. The molecule has C_i symmetry. The angle between each of the pyridyl rings and the perylene-imide skeleton is 74.5 (2)°. The molecules are stacked along the *c* axis with a tilt angle of about 31.5° between adjacent molecules.

Comment

Perylene compounds are industrially important pigments, covering a variety of shades from red *via* maroon to black (Herbst & Hunger, 1993). The title compound, (I), is a *para*-pyridyl derivative, abbreviated to PPP.



(I)

We have carried out a series of investigations on H_2 gas sensors utilizing a high proton affinity of organic pigments that have pyridyl rings connected directly to the choromophore (Takahashi & Mizuguchi, 2005). The N atom of the pyridyl ring works as a strong proton acceptor and plays a decisive role for signal detection. PPP exhibits a resistivity change of about three orders of magnitude even for 0.05% H_2. Other than the present *para* derivative, there are also *ortho* and *meta*-derivatives and their sensitivity is slightly different, depending the site of the N atom. In this connection, structure analyses of these derivatives have been carried out. The structures of the *ortho* and *meta* derivatives are reported in the preceding two papers (Mizuguchi *et al.*, 2005*a*,*b*). This paper reports the structure of the *para* derivative.

The molecule of PPP (Fig. 1) is characterized by C_i symmetry. The angle between each of the pyridyl rings and the perylene-imide skeleton is 74.5 (2)°. The perylene–imide skeleton is planar (r.m.s. deviation = 0.032 Å). The molecules are stacked along the *c* axis with a tilt angle of 31.5° between adjacent molecules, as shown in Fig. 2.

Experimental

PPP was synthesized by reaction of perylenetetracarboxylic dianhydride with 1,2-di-4-pyridyldiamine in dimethylnaphthalene at 490 K for 3 h according to the method of Herbst & Hunger (1993). The product was then purified three times by sublimation at 760 K, using a two-zone furnace (Mizuguchi, 1981). Single crystals of PPP were grown from a solution in 1-chloronaphthalene After 72 h, a number of single crystals were obtained in the form of needles.

Crystal data

$C_{34}H_{16}N_4O_4$
M_r = 544.51
Orthorhombic, *Pccn*
a = 21.232 (2) Å
b = 15.890 (2) Å
c = 6.9311 (8) Å
V = 2338.4 (5) Å^3
Z = 4
D_x = 1.547 Mg m^{-3}
Cu Kα radiation
Cell parameters from 8331 reflections
θ = 3.5–68.2°
μ = 0.85 mm^{-1}
T = 93.2 K
Needle, red
0.25 × 0.05 × 0.05 mm

Data collection

Rigaku R-AXIS RAPID Imaging Plate diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.554, T_{max} = 0.958
18572 measured reflections
1965 independent reflections
839 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.077
θ_{max} = 68.2°
$h = -24 \rightarrow 23$
$k = -18 \rightarrow 18$
$l = -7 \rightarrow 6$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.148
S = 1.09
1965 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2)]$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.211 (5)	C5—C6	1.429 (6)
O2—C11	1.231 (5)	C5—C7^{i}	1.466 (6)
N1—C1	1.415 (6)	C6—C7	1.424 (5)
N1—C11	1.402 (5)	C6—C12	1.426 (6)
C1—C2	1.459 (6)	C7—C8	1.401 (6)
C2—C3	1.386 (6)	C8—C9	1.397 (6)
C2—C12	1.415 (6)	C9—C10	1.373 (6)
C3—C4	1.380 (6)	C10—C11	1.476 (6)
C4—C5	1.399 (6)	C10—C12	1.408 (6)
C1—N1—C11	124.4 (4)	C5^{i}—C7—C6	119.8 (4)
O1—C1—N1	119.4 (4)	C5^{i}—C7—C8	121.9 (4)
O1—C1—C2	123.5 (5)	C6—C7—C8	118.3 (4)
N1—C1—C2	117.1 (4)	C7—C8—C9	121.9 (4)
C1—C2—C3	119.0 (4)	C8—C9—C10	120.5 (4)
C1—C2—C12	120.8 (4)	C9—C10—C11	119.3 (4)
C3—C2—C12	120.2 (4)	C9—C10—C12	119.7 (4)
C2—C3—C4	120.1 (4)	C11—C10—C12	121.0 (4)
C3—C4—C5	122.7 (4)	O2—C11—N1	120.2 (4)
C4—C5—C6	117.7 (4)	O2—C11—C10	123.2 (4)
C4—C5—C7^{i}	123.2 (4)	N1—C11—C10	116.6 (4)
C6—C5—C7^{i}	119.0 (4)	C2—C12—C6	119.4 (4)
C5—C6—C7	121.2 (4)	C2—C12—C10	120.0 (4)
C5—C6—C12	119.8 (4)	C6—C12—C10	120.6 (4)
C7—C6—C12	119.0 (4)		

Symmetry code: (i) $\frac{3}{2} - x, \frac{3}{2} - y, z$.

All H atoms were positioned geometrically [C—H = 0.95 Å and $U_{iso} = 1.2U_{eq}$(C)] and refined using a riding model.

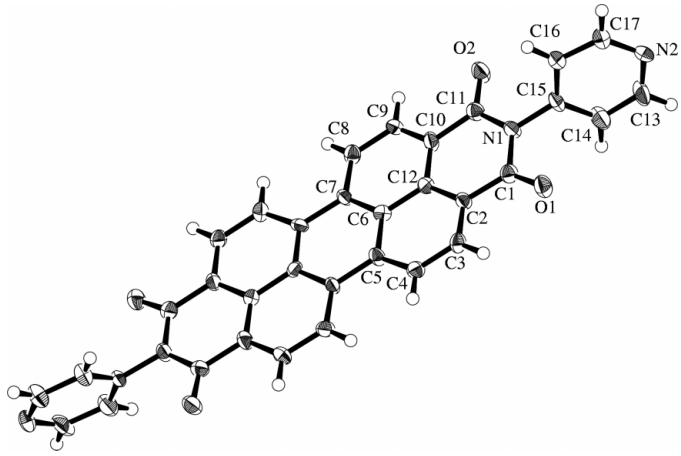


Figure 1
A view of the molecular conformation of (I), showing 50% displacement ellipsoids for the non-H atoms. Unlabelled atoms are related to labelled atoms by $\frac{3}{2} - x, \frac{3}{2} - y, z$.

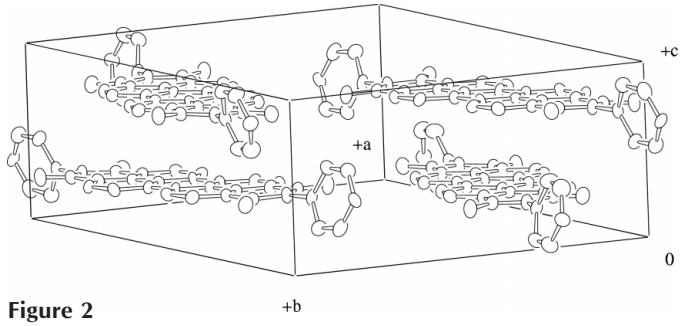


Figure 2
The packing arrangement of PPP.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *TEXSAN*; molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *TEXSAN*.

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