Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

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

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) = 0.004 Å
R factor = 0.039
wR factor = 0.073
Data-to-parameter ratio = 10.1

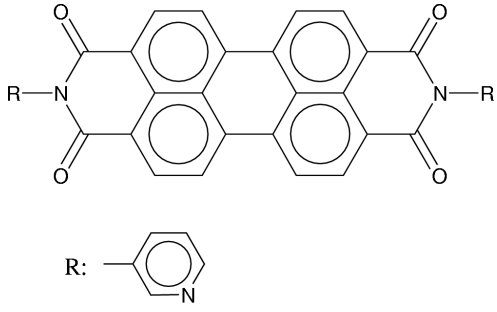
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 14 January 2005
Accepted 19 January 2005
Online 29 January 2005

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 with one half-molecule in the asymmetric unit. The angle between each of the pyridyl rings and the perylene-imide skeleton is 54.88 (10)°. The molecules are stacked in a 'hunter's fence' fashion (*viz.* when viewed from the side, molecules, slipped by 45° within molecular stacks, cross each other in a fence-like structure) along the *b* axis.

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 *meta*-pyridyl derivative, abbreviated to MPP.



(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 acts as a strong proton acceptor and plays a decisive role for signal detection. MPP exhibits a resistivity change of about three orders of magnitude, even for 0.05% H_2. Other than the present *meta* derivative, there are also *ortho* and *para* derivatives and their sensitivity is slightly different, depending on the site of the N atom. In this connection, structure analyses of these derivatives have been carried out. The structures of *ortho* and *para* derivatives are reported in the following papers (Mizuguchi *et al.*, 2005; Hino *et al.*, 2005). This paper reports the structure of the *meta* derivative.

The molecule of MPP (Fig. 1) is characterized by C_i symmetry. The angle between each of the pyridyl rings and the perylene-imide skeleton is 54.88 (10)°. The perylene–imide skeleton is planar (r.m.s. deviation = 0.035 Å). The molecules are stacked in a 'hunter's fence' fashion (*viz.* when viewed from the side, molecules, slipped by 45° within molecular stacks, cross each other in a fence-like structure) along the *b* axis, as shown in Fig. 2.

Experimental

MPP was synthesized by reaction of perylenetetracarboxylic dianhydride with 1,2-di-3-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 MPP were grown from the vapor phase in a closed system based on a two-zone furnace. After 48 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
Monoclinic, $P2_1/n$
a = 15.422 (2) Å
b = 3.8275 (6) Å
c = 19.282 (3) Å
β = 103.29 (1)°
V = 1107.7 (3) Å^3
Z = 2
D_x = 1.633 Mg m^{-3}
Cu Kα radiation
Cell parameters from 6312 reflections
θ = 3.2–65.5°
μ = 0.90 mm^{-1}
T = 93.2 K
Needle, red
0.50 × 0.07 × 0.03 mm

Data collection

Rigaku R-AXIS RAPID Imaging Plate diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.973, T_{max} = 0.973
10013 measured reflections
1922 independent reflections
1009 reflections with $F^2 > 2\sigma(F^2)$
R_{int} = 0.048
θ_{max} = 68.2°
$h = -18 \rightarrow 18$
$k = -4 \rightarrow 4$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.073
S = 0.94
1922 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + \{0.025[\mathrm{Max}(F_o^2,0) + 2F_c^2]/3\}^2]$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.222 (3)	C5—C6	1.433 (3)
O2—C11	1.218 (3)	C5—C7^{i}	1.470 (3)
N1—C1	1.414 (3)	C6—C7	1.431 (3)
N1—C11	1.411 (3)	C6—C12	1.418 (3)
C1—C2	1.475 (3)	C7—C8	1.394 (3)
C2—C3	1.381 (3)	C8—C9	1.393 (3)
C2—C12	1.407 (3)	C9—C10	1.379 (3)
C3—C4	1.396 (3)	C10—C11	1.477 (3)
C4—C5	1.383 (3)	C10—C12	1.413 (3)
C1—N1—C11	124.1 (2)	C5^{i}—C7—C6	119.1 (2)
O1—C1—N1	120.5 (2)	C5^{i}—C7—C8	122.0 (2)
O1—C1—C2	122.6 (2)	C6—C7—C8	118.9 (2)
N1—C1—C2	116.9 (2)	C7—C8—C9	121.1 (2)
C1—C2—C3	118.7 (2)	C8—C9—C10	121.1 (2)
C1—C2—C12	120.9 (2)	C9—C10—C11	119.4 (2)
C3—C2—C12	120.4 (2)	C9—C10—C12	119.5 (2)
C2—C3—C4	119.9 (2)	C11—C10—C12	121.0 (2)
C3—C4—C5	122.2 (2)	O2—C11—N1	120.6 (2)
C4—C5—C6	118.4 (3)	O2—C11—C10	122.7 (3)
C4—C5—C7^{i}	122.2 (2)	N1—C11—C10	116.6 (2)
C6—C5—C7^{i}	119.4 (2)	C2—C12—C6	119.8 (2)
C5—C6—C7	121.5 (2)	C2—C12—C10	120.0 (3)
C5—C6—C12	119.4 (3)	C6—C12—C10	120.2 (2)
C7—C6—C12	119.1 (2)		

Symmetry code: (i) $1-x, 1-y, -z$.

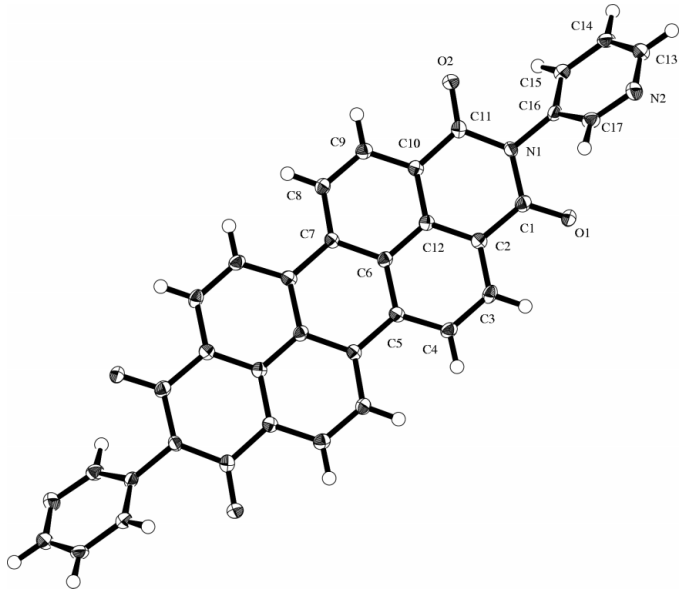


Figure 1
A view of the molecular conformation of (I), showing 50% displacement ellipsoids for the non-H atoms. Unlabelled atoms are related to labelled atoms by $1-x, 1-y, -z$

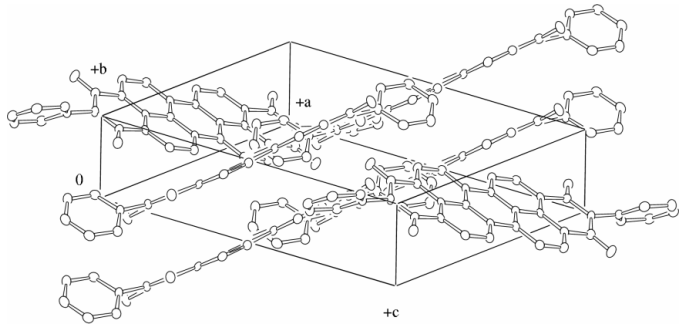


Figure 2
The packing arrangement of MPP

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

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, 198); program(s) used to refine structure: *TEXSAN*; molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *TEXSAN*.

The authors are indebted to Mr I. Suzuki for experimental assistance.

References

Burnett, M. N. & Johnson, C. K.(1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory. Tennessee, USA.
Herbst, W. & Hunger, K. (1993). *Industrial Organic Pigments*, pp. 467–475. Weinheim: VCH.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Hino, K., Sato, K., Takahashi, H., Suzuki, S. & Mizuguchi, J. (2005). *Acta Cryst.* E**61**, o440–o441.

Mizuguchi, J. (1981). *Cryst. Res. Technol.* **16**, 695–700.
Mizuguchi, J., Hino, K., Sato, K., Takahashi, H. & Suzuki, S. (2005). *Acta Cryst.* E**61**, o437–o439.
Molecular Structure Corporation (2001). *TEXSAN*. Version 1.11. MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (1985). *SHELXS86*. *Crystallographic Computing 3*, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford University Press.
Takahashi, H. & Mizuguchi, J. (2005). *J. Electrochem. Soc.* In the press.