$\omega/2\theta$ scans
Absorption correction: none
4473 measured reflections
4325 independent reflections
3016 reflections with $I > 2\sigma(I)$

$h = -8 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -14 \rightarrow 14$
3 standard reflections
frequency: 60 min
intensity decay: 1.1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.122$
$S = 1.049$
4325 reflections
314 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0579P)^2 + 0.0076P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.282$ e Å^{-3}
$\Delta\rho_{\min} = -0.211$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

N—C17	1.485 (4)	O4—C18	1.240 (4)
N—C16	1.490 (4)	O5—C18	1.255 (4)
N—C9	1.515 (4)		
C17—N—C16	111.1 (2)	C17—N—H1	102 (2)
C17—N—C9	114.0 (2)	C16—N—H1	109 (2)
C16—N—C9	112.8 (2)	C9—N—H1	107 (2)
O4—C18—C19—C20	−118.2 (3)	O6—C20—C21—C22	117.5 (3)
O5—C18—C19—C20	62.1 (4)	C19—C20—C21—C22	−117.3 (3)
C18—C19—C20—O6	175.6 (3)	O6—C20—C21—C26	−63.1 (4)
C18—C19—C20—C21	49.1 (4)	C19—C20—C21—C26	62.1 (4)

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···A	D—H	H···A	D···A	D—H···A
N—H1···O4$^{\text{i}}$	0.91 (4)	1.72 (4)	2.620 (3)	171 (3)
O1—H2···O5	0.82 (4)	1.74 (4)	2.535 (3)	166 (4)
O3—H3···O6	0.84 (4)	1.99 (4)	2.775 (4)	156 (4)
O6—H21···O1$^{\text{ii}}$	0.89 (4)	1.98 (5)	2.820 (4)	155 (4)

Symmetry codes: (i) $x, y-1, 1+z$; (ii) $x-1, y, z$.

The enantiomorph was chosen with respect to the known stereochemistry of (−)-morphine. R_{int} is based on only 148 reflections; if Friedel pairs are merged, $R_{\text{int}} = 0.0495$. All H atoms were located in a difference electron-density map. The positions of the ammonium and hydroxy H atoms were allowed to refine with individual isotropic displacement parameters. All other H atoms were constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ [$1.5U_{\text{eq}}(\text{C})$ for methyl H atoms].

Data collection: locally modified *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *SET*4 (de Boer & Duisenberg, 1984). Data reduction: *HELENA* (Spek, 1997). Program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1997). Program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997). Molecular graphics: *PLATON* (Spek, 1998). Software used to prepare material for publication: *PLATON*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1054). Services for accessing these data are described at the back of the journal.

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N,*N'*-Bis(2-phenethyl)perylene-3,4:9,10-bis(dicarboximide)

Jin Mizuguchi

Department of Applied Physics, Faculty of Engineering, Yokohama National University, 79-5 Tokiwadai, Hodogaya-ku, 240-8501 Yokohama, Japan. E-mail: mizuj@ed.ynu.ac.jp

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Abstract

The molecule of the title compound, $C_{40}H_{26}N_2O_4$, belongs to point group C_i. The perylene ring system is entirely planar, but is not fully delocalized, as shown by the fact that some C—C bonds are significantly longer compared with those of normal aromatic compounds. The phenyl rings at both ends of the molecule are not completely parallel to the plane of the perylene skeleton, but are slightly twisted in the same direction by about 2.7 (2)°.

Comment

N,*N'*-Bis(2-phenethyl)perylene-3,4:9,10-bis(dicarboximide), (I), is a commercial black pigment based on the

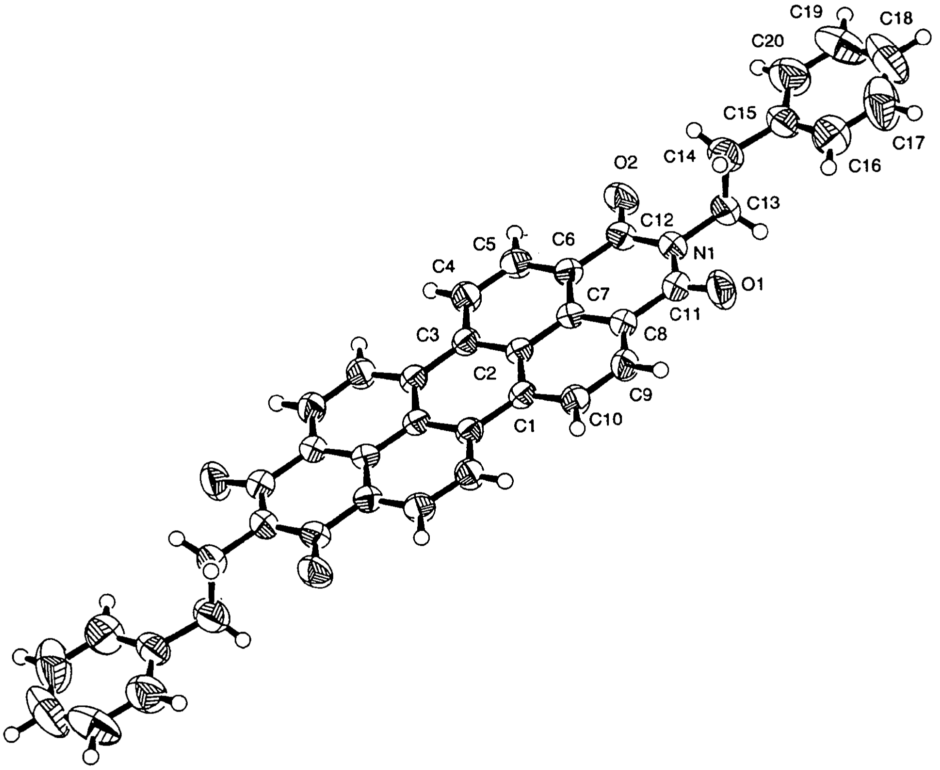
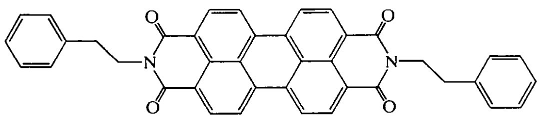


Fig. 1. *ORTEP*II (Johnson, 1976) plot of PB showing the numbering of the non-H atoms. Displacement ellipsoids are scaled to the 50% probability level.

perylene skeleton ('Paliogen Black L0084', BASF AG; hereafter abbreviated to PB) (Herbst & Hunger, 1993). This compound can also be utilized as a photoconductor for copiers (Duff *et al.*, 1990). To our surprise, PB exhibits a brilliant red color (absorption maximum: 500 nm) as it is evaporated, but it undergoes a color change from red to black (absorption maxima: 473 and 610 nm) when exposed to acetone vapor or when heated above 373 K for several seconds. We then utilized the present color change from red (amorphous) to black (crystalline), or *vice versa*, for optical disks based on a GaInAlP diode laser of 635 nm (Mizuguchi, 1997*a*,*b*). In the course of our studies on the electronic characterization of (I), we found a different crystalline phase from the one reported by Hädicke & Graser (1986), as obtained by recrystallization from cresol.



(I)

The title molecule belongs to point group C_i (Fig. 1). The perylene ring system is entirely planar, but is not fully delocalized. In particular, the bond lengths C1—C3$^{\mathrm{i}}$ [1.461 (4) Å], C8—C11 [1.477 (4) Å] and C6—C12 [1.480 (4) Å] are significantly longer compared with those of normal aromatic compounds [symmetry code: (i) $1-x$, $1-y$, $2-z$], whereas the C—C bond lengths in two adjacent phenyl rings (C2–C7 and C1, C2, C7–C10) correspond to those of naphthalene (Bondi, 1964). A packing diagram of PB showing a side view of the molecule is presented in Fig. 2. The phenyl rings at both ends of the molecule are not completely parallel to the perylene skeleton and are slightly twisted in the same direction by about 2.7 (2)°. The molecules are arranged in a zigzag fashion along the *b* axis.

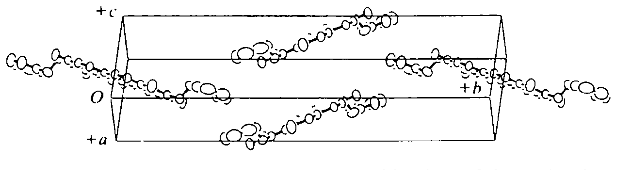


Fig. 2. Packing diagram of PB showing a side view of the molecule.

Details on the correlation between the crystal structure and optical properties are reported elsewhere (Mizuguchi, 1998).

Experimental

PB was refined four times by sublimation in a two-zone furnace (Mizuguchi, 1981). Single crystals were then grown from the vapor phase using argon as the carrier gas in the same sublimation equipment. PB was sublimated at about 713 K, and the argon flow rate was controlled to 20 $\text{mm}^3\ \text{s}^{-1}$ using a mass flow meter (F-100/200. Bronkhorst BV). After 24 h of vapor growth, a number of platelet-like single crystals were obtained in the sublimation tube. The density of PB was measured by flotation in benzene/carbontetrachloride.

Crystal data

$C_{40}H_{26}N_2O_4$
$M_r = 598.66$
Monoclinic
$P2_1/c$
$a = 4.737\ (1)$ Å
$b = 32.450\ (3)$ Å
$c = 9.507\ (1)$ Å
$\beta = 100.27\ (1)°$
$V = 1438.0\ (1)$ Å^3
$Z = 2$
$D_x = 1.383$ Mg m^{-3}
$D_m = 1.342$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 10.0–17.5°$
$\mu = 0.721$ mm^{-1}
$T = 298.2$ K
Acicular
0.54 × 0.12 × 0.07 mm
Black

Data collection

Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3385 measured reflections
3109 independent reflections
1880 reflections with $I > \sigma(I)$

$R_{\text{int}} = 0.017$
$\theta_{\text{max}} = 75°$
$h = 0 \rightarrow 5$
$k = 0 \rightarrow 40$
$l = -11 \rightarrow 11$
3 standard reflections
frequency: 60 min
intensity decay: 1.04%

Refinement

Refinement on F
$R = 0.076$
$wR = 0.109$
$S = 1.340$
1880 reflections
209 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o) + 0.00319|F_o|^2]$
$(\Delta/\sigma)_{\text{max}} < 0.0001$

$\Delta\rho_{\text{max}} = 0.25$ e Å^{-3}
$\Delta\rho_{\text{min}} = -0.22$ e Å^{-3}
Extinction correction: type 2 Gaussian isotropic (Zachariasen, 1967)
Extinction coefficient: 0.01 (2)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

O1—C11	1.213 (4)	C6—C12	1.480 (4)
O2—C12	1.206 (4)	C7—C8	1.412 (4)
N1—C11	1.397 (4)	C8—C9	1.374 (5)
N1—C12	1.401 (4)	C8—C11	1.477 (4)
N1—C13	1.479 (4)	C9—C10	1.388 (4)
C1—C2	1.425 (4)	C13—C14	1.503 (6)
C1—C3^i	1.461 (4)	C14—C15	1.513 (5)
C1—C10	1.392 (4)	C15—C16	1.367 (6)
C2—C3	1.429 (4)	C15—C20	1.375 (6)
C2—C7	1.425 (4)	C16—C17	1.343 (7)
C3—C4	1.397 (4)	C17—C18	1.37 (1)
C4—C5	1.380 (4)	C18—C19	1.35 (1)
C5—C6	1.372 (4)	C19—C20	1.360 (8)
C6—C7	1.415 (4)		
C11—N1—C12	125.0 (3)	C7—C8—C11	120.4 (3)
C11—N1—C13	117.3 (3)	C9—C8—C11	119.6 (3)
C12—N1—C13	117.6 (3)	C8—C9—C10	120.4 (3)
C2—C1—C3^i	119.0 (2)	C1—C10—C9	122.0 (3)
C2—C1—C10	118.6 (2)	O1—C11—N1	120.2 (3)
C3^i—C1—C10	122.5 (3)	O1—C11—C8	122.7 (3)
C1—C2—C3	121.8 (2)	N1—C11—C8	117.1 (3)
C1—C2—C7	119.0 (2)	O2—C12—N1	120.7 (3)
C3—C2—C7	119.1 (2)	O2—C12—C6	122.7 (3)
C1^i—C3—C2	119.2 (2)	N1—C12—C6	116.6 (3)
C1^i—C3—C4	122.5 (2)	N1—C13—C14	111.3 (3)
C2—C3—C4	118.3 (2)	C13—C14—C15	111.3 (3)
C3—C4—C5	121.8 (3)	C14—C15—C16	120.8 (4)
C4—C5—C6	121.1 (3)	C14—C15—C20	121.1 (4)
C5—C6—C7	119.7 (3)	C16—C15—C20	118.0 (4)
C5—C6—C12	119.7 (3)	C15—C16—C17	120.7 (5)
C7—C6—C12	120.6 (3)	C16—C17—C18	120.6 (5)
C2—C7—C6	119.9 (2)	C17—C18—C19	119.7 (6)
C2—C7—C8	119.9 (3)	C18—C19—C20	119.8 (6)
C6—C7—C8	120.2 (3)	C15—C20—C19	121.0 (5)
C7—C8—C9	120.0 (3)		

Symmetry code: (i) $1 - x, 1 - y, 2 - z$.

A polarization microscope clearly showed that the crystal analyzed was a 1:1 twin so that the $0kl$ reflections due to both twin lattices coincide. A twin correction (removal of the redundant $0kl$ reflections) was therefore made for the structure analysis. This greatly improved the R value, but it still remained around 0.076. The non-H atoms were refined anisotropically by full-matrix least-squares methods. H atoms were included but not refined.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *TEXSAN*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1058). Services for accessing these data are described at the back of the journal.

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