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Dibenzimidazo[2,1-*a*:2',1'-*a'*]anthra[2,1,9-*def*:6,5,10-*d'e'f'*]diisoquinoline-10,21-dione: *trans* form (I)

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Dibenzimidazo[2,1-*a*:2',1'-*a'*]anthra-[2,1,9-*def*:6,5,10-*d'e'f'*]diisoquinoline-10,21-dione: *trans* form (I)**Jin Mizuguchi**

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Key indicators

Single-crystal X-ray study

$T = 93\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.012\text{ \AA}$

R factor = 0.102

wR factor = 0.222

Data-to-parameter ratio = 9.5

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

The title compound, $\text{C}_{36}\text{H}_{16}\text{N}_4\text{O}_2$, is the *trans* form of a benzimidazole perylene derivative used as a black pigment. The molecule is entirely planar and possesses a center of symmetry. The molecules are oriented in the same direction and are stacked with a considerable overlap of the perylene imide skeleton along the *a* axis.

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Comment

Perylene and perinone compounds are well known organic pigments which exhibit shades in the range from red to black (Herbst & Hunger, 1993). Both are chemically related. Perylene compounds are derived from perylene-3,4,9,10-tetracarboxylic acid, while perinones are derivatives of naphthalene-1,4,5,8-tetracarboxylic acid. The title compound, (I), is the *trans* form of a benzimidazole perylene derivative (abbreviated to BIP) which has attracted attention as a black pigment (Mizuguchi & Shimo, 2005), as well as an electronic material for electroluminescence (EL) devices (Adachi *et al.*, 1988) and electrophotographic photoreceptors (Loutfy *et al.*, 1989). In perinone analogues of BIP, there are *cis* and *trans* isomers, known as Pigment Orange 43 and Pigment Red 194, on the market, respectively. These structures have recently been reported (Mizuguchi, 2003*a,b*). On the other hand, no structure of BIP has been reported, although the structural information is quite important for electronic applications. The present paper deals with the crystal structure of the *trans* form (I) of BIP while the report on the *cis* form (II) is in the following paper (Mizuguchi, 2005).

The structure of the *trans* form (I) [$P\bar{1}$; $a = 4.729(2)\text{ \AA}$, $b = 8.282(2)\text{ \AA}$, $c = 14.693(4)\text{ \AA}$, $\alpha = 89.35(2)^\circ$, $\beta = 91.15(3)^\circ$ and $\gamma = 105.83(3)^\circ$] is similar in most lattice parameters to those of the *cis* form (II) [$P2_1/c$; $a = 4.7501(6)\text{ \AA}$, $b = 28.079(3)\text{ \AA}$, $c = 8.728(1)\text{ \AA}$ and $\beta = 99.21(1)^\circ$], but the former cell volume is approximately half of the latter. An *ORTEP* plot (Burnett & Johnson, 1996) of (I) is shown in Fig. 1. The molecule is entirely planar and possesses a center of symmetry. All molecules are oriented in the same direction and are stacked with

a considerable overlap of the perylene imide skeleton along the *a* axis, as shown in Fig. 2.

Experimental

BIP was synthesized by reaction of perylenetetracarboxylic dianhydride with 1,2-phenylenediamine in phenol at 483 K for 6 h (Tamizhmani *et al.*, 1991). The products contained both *trans*-(I) and *cis*-(II) isomers of BIP. The isomers were separated by chromatography using a carrier based on a 1:5 mixed solvent of trifluoroacetic acid and toluene. The *cis* or *trans* configuration has been confirmed by means of UV-vis solution spectra together with molecular orbital calculations. BIP powders of the *trans* form (I) were purified by sublimation under argon at about 675 K, using a two-zone furnace (Mizuguchi, 1981). Single crystals were then grown from the vapor phase in a closed system at about 680 K. After 48 h, a number of black needle-shaped crystals of (I) were obtained, but these were slightly curved.

Crystal data

$C_{36}H_{16}N_4O_2$	$Z = 1$
$M_r = 536.53$	$D_x = 1.602 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Cu $K\alpha$ radiation
$a = 4.729 \text{ (2) \AA}$	Cell parameters from 3329 reflections
$b = 8.282 \text{ (2) \AA}$	$\theta = 3.0\text{--}68.2^\circ$
$c = 14.693 \text{ (4) \AA}$	$\mu = 0.82 \text{ mm}^{-1}$
$\alpha = 89.35 \text{ (2)^\circ}$	$T = 93.2 \text{ K}$
$\beta = 91.15 \text{ (3)^\circ}$	Needle, black
$\gamma = 104.83 \text{ (3)^\circ}$	$0.40 \times 0.05 \times 0.05 \text{ mm}$
$V = 556.2 \text{ (3) \AA}^3$	

Data collection

Rigaku R-Axis RAPID Imaging	1831 independent reflections
Plate diffractometer	505 reflections with $F^2 > 2\sigma(F^2)$
ω scans	$R_{\text{int}} = 0.106$
Absorption correction: multi-scan	$\theta_{\text{max}} = 68.2^\circ$
(<i>ABSCOR</i> ; Higashi, 1995)	$h = -4 \rightarrow 4$
$T_{\text{min}} = 0.490$, $T_{\text{max}} = 0.960$	$k = -9 \rightarrow 9$
5005 measured reflections	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	H-atom parameters not refined
$R[F^2 > 2\sigma(F^2)] = 0.102$	$w = 1/[\sigma^2(F_o^2) + \{0.042[\max(F_o^2, 0) + 2F_c^2/3]\}]$
$wR(F^2) = 0.222$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.93$	$\Delta\rho_{\text{max}} = 0.41 \text{ e \AA}^{-3}$
1804 reflections	$\Delta\rho_{\text{min}} = -0.41 \text{ e \AA}^{-3}$
190 parameters	

Table 1

Selected geometric parameters ($\text{\AA}$, $^\circ$).

N1—C11	1.41 (1)	N2—C13	1.42 (1)
N1—C18	1.39 (1)	C13—C18	1.41 (1)
N2—C11	1.30 (1)		
C4—C5—C7 ⁱ —C8 ⁱ	0 (1)		

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

All H atoms were positioned geometrically [$C-H = 0.95 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$] and not refined. Since the single crystal needle was slightly curved and the crystallinity was rather poor, the final *R* factor (0.102) remained rather high.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *TEXSAN* (Molecular

Figure 1

A view of the molecular structure of (I), showing 50% displacement ellipsoids for non-H atoms. Unlabeled atoms are related to labeled atoms by the operation $(-x, 1 - y, -z)$.

Figure 2

The molecular arrangement of (I) in the crystal structure.

Structure Corporation, 2001); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); 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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