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4-[4-(1,3-Dioxo-2,3-dihydro-1*H*-isoindol-2-yl)-butoxy]phthalonitrile

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.002 Å
R factor = 0.041
wR factor = 0.108
Data-to-parameter ratio = 13.1

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

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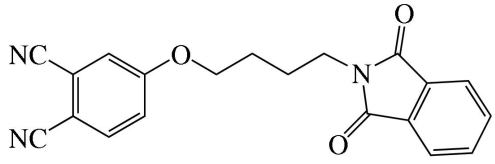
The title compound, $C_{20}H_{15}N_3O_3$, consists a phthalonitrile group and an isoindole-1,3-dione group connected by a flexible butoxy chain. The dihedral angle between these two groups is 70.47 (3)°. Partial face-to-face overlap between the phthalonitrile groups of centrosymmetrically related molecules is observed. In addition, the crystal packing is stabilized by C—H···O hydrogen bonds.

Comment

The title compound, (I), can be used as a precursor in the synthesis of phthalimide-substituted phthalocyanines. The phthalimide groups can be converted to amino-substituted phthalocyanines by treatment with hydrazine at room temperature (Fernandez *et al.*, 1995). This precursor can be condensed with other hydrophobic precursors to form amphiphilic phthalocyanines, which seem to have better interaction with cell tissues in the photodynamic therapy (PDT) of tumors (Griffiths *et al.*, 1994).



(I)

The molecular structure of (I) is shown in Fig. 1. The N1—C7 [1.1386 (17) Å] and N2—C8 [1.1375 (17) Å] bond lengths show normal values (Nesi *et al.*, 1998). The C1—C7 [1.4389 (17) Å] and C2—C8 [1.4391 (17) Å] bond distances are comparable to the mean value of 1.443 (8) Å reported for Csp^2—Csp^1 bonds by Allen *et al.* (1987). The dihedral angle between the C1—C6 benzene ring and the isoindole group is 70.47 (3)°.

In the crystal packing, the molecules are stacked along the short *a* axis, without any π–π interaction. However, partial face-to-face overlap between the C1–C5 benzene rings of the molecules at (*x*, *y*, *z*) and (−*x*, 1 − *y*, −*z*) is observed, with a perpendicular distance of 3.425 Å. In addition, the molecular packing is stabilized by C—H···O hydrogen bonds (Table 1).

Experimental

3-Hydroxyphthalonitrile and *N*-(4-bromobutyl)phthalimide were synthesized according to the literature procedures of Li *et al.* (1996) and Kuang *et al.* (2001), respectively. 3-Hydroxyphthalonitrile (1.5 g, 10 mmol) and *N*-(4-bromobutyl)phthalimide (4 g, 14 mmol) were dissolved in anhydrous dimethylformamide (150 ml) under an N_2 atmosphere, and then dry fine-powdered potassium carbonate (5.5 g)

was added with vigorous stirring. The reaction mixture was stirred for 6 h at 333 K and then poured into ice-water. The filter cake was then recrystallized from tetrahydrofuran and dried. Single crystals of (I) (yield 40%) were obtained *via* slow evaporation of a solution in absolute tetrahydrofuran at room temperature. IR (KBr, cm^{-1}): ν 3045 (Ar—CH), 2221(C—N), 2881, 2956 (C—H), 1704 (C—O), 1597, 1498, 1469, 1435, 1396, 1374, 1356, 1323, 1171, 1136, 1085, 1054, 1004, 925, 884, 853, 793, 757, 720, 528. ^{1}H NMR ($CDCl_3$, p.p.m.): δ 1.754 (*s*, 4H), 3.641 (*s*, 2H), 4.166 (*s*, 2H), 7.417–7.435 (*m*, 1H), 7.734–7.738 (*d*, 1H), 7.844–7.857 (*m*, 4H), 8.009–8.027 (*d*, 1H).

Crystal data

$C_{20}H_{15}N_3O_3$	D_x = 1.351 Mg m^{-3}
M_r = 345.35	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 3884 reflections
a = 5.8020 (3) Å	θ = 3.1–27.5°
b = 13.6911 (8) Å	μ = 0.09 mm^{-1}
c = 21.4006 (14) Å	T = 298 (2) K
β = 92.721 (4)°	Prism, colorless
V = 1698.06 (17) Å^3	0.75 × 0.52 × 0.35 mm
Z = 4	

Data collection

Rigaku Mercury CCD area-detector diffractometer	3327 reflections with $I > 2\sigma(I)$
	R_{int} = 0.021
ω scans	θ_{max} = 27.5°
Absorption correction: none	$h = -7 \rightarrow 7$
12 870 measured reflections	$k = -17 \rightarrow 17$
3876 independent reflections	$l = -27 \rightarrow 25$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.2969P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.041	
$wR(F^2)$ = 0.108	
S = 1.01	$(\Delta/\sigma)_{max}$ = 0.001
3876 reflections	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
295 parameters	$\Delta\rho_{min}$ = −0.14 e Å^{-3}
All H-atom parameters refined	

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H1···O2^{i}	0.969 (15)	2.327 (15)	3.2766 (16)	166.3 (12)
C6—H3···O3^{ii}	0.963 (14)	2.418 (14)	3.3215 (16)	156.0 (11)
C9—H4···O2	0.999 (15)	2.590 (15)	3.4098 (18)	139.3 (11)
C11—H9···O2^{iii}	0.986 (16)	2.465 (15)	3.1902 (17)	130.1 (11)
C12—H10···O2	0.994 (15)	2.466 (14)	2.8946 (18)	105.5 (10)

Symmetry codes: (i) $-x, 1-y, -z$; (ii) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iii) $1+x, y, z$.

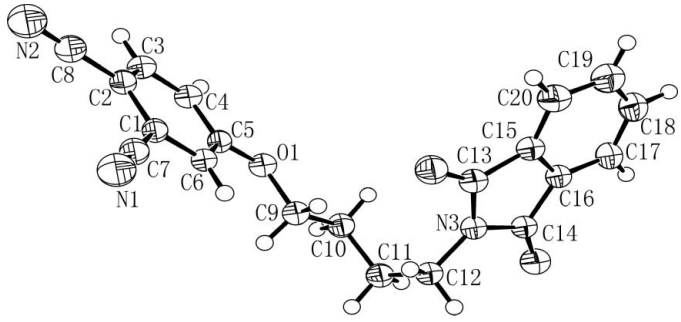


Figure 1
The structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

All H atoms were located in a difference Fourier map and refined isotropically. The C—H distances lie in the range 0.964 (15)–1.010 (16) Å.

Data collection: *CrystalClear* (Rigaku, 2002); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *SHELXL97*.

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