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Dimethyl 5-(3,4-dicyanophenoxy)isophthalate

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.113
Data-to-parameter ratio = 18.5

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

The crystal structure of the title compound, $C_{18}H_{12}N_2O_5$, is stabilized by intermolecular C—H···O and π–π interactions.

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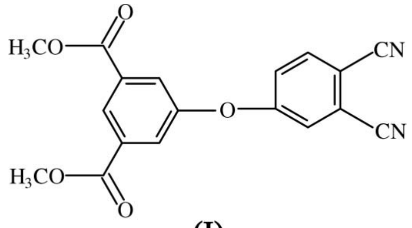
Comment

5-(3,4-Dicyanophenoxy)dimethyl isophthalate is a starting material in the synthesis of tetrasubstituted dendritic phthalocyanines (McKeown, 1998; Kimura *et al.*, 1997). In addition to their extensive use as dyes and pigments, phthalocyanines have found widespread applications in catalysis, optical recording, photoconductive materials and photodynamic therapy, and as chemical sensors (Leznoff & Lever, 1989–1996).



(I)

The molecular structure of (I) is shown in Fig. 1. The cyano group bond lengths (Table 1) are short enough to indicate their triple-bond character and agree with literature values (Atalay *et al.*, 2004).

The phthalonitrile and C9–C14 rings are each nearly planar and the dihedral angle between them is 67.58 (7)°. The bond lengths and angles of the ester groups in the dimethyl isophthalate fragment are similar to values reported by Ocak *et al.* (2004).

There are two intermolecular C—H···O hydrogen bonds in the crystal structure of (I), and the crystal packing is further stabilized by π–π stacking interactions [perpendicular distance 3.7139 (7) Å] involving the phthalonitrile ring C2–C7 and the C2–C7 ring of the symmetry-related molecule at $(-x, 1-y, 1-z)$.

Experimental

5-Hydroxyisophthalic acid (1.30 g, 6.19 mmol) and 4-nitrophthalonitrile (1.00 g, 5.78 mmol) were dissolved in dry dimethylormamide (40 ml) with stirring under N_2. Dry fine-powdered potassium carbonate (1.5 g, 10.87 mmol) was added in portions (10 × 1 mmol) every 10 min. The reaction mixture was stirred for 48 h at 333 K and poured into ice–water (200 g). The product was filtered off and washed with NaOH solution (10% *w*/*w*) and water until the filtrate was neutral. Recrystallization from ethanol gave a white product (yield 0.74 g, 38.54%). Single crystals of (I) were obtained from a solution in dry dimethylformamide at room temperature *via* slow evaporation (m.p. 422–425 K). IR (ν_{max}, cm^{-1}): 3127–3048 (Ar—CH$_2$), 2861 (CH$_2$), 2241 (CN), 1739 (C═O), 1590, 1499, 1441, 1391, 1317, 1259, 1210, 1160, 1110, 1011, 987, 961, 928, 903, 845, 796, 763.

Crystal data

$C_{18}H_{12}N_2O_5$
M_r = 336.30
Monoclinic, $P2_1/n$
a = 13.2925 (8) Å
b = 7.2911 (3) Å
c = 16.7695 (10) Å
β = 94.894 (5)°
V = 1619.32 (15) Å^3

Z = 4
D_x = 1.379 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
Prism, yellow
0.52 × 0.32 × 0.24 mm

Data collection

Stoe IPDS II diffractometer
ω scans
Absorption correction: none
29194 measured reflections

4206 independent reflections
2709 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 28.8°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.113
S = 1.03
4206 reflections
227 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0621P)^2 + 0.0155P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.017 (2)

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.1373 (18)	C15—O3	1.3239 (16)
C5—O1	1.3758 (13)	C16—O4	1.1984 (15)
C8—N2	1.1378 (19)	C16—O5	1.3240 (14)
C9—O1	1.3943 (13)	C17—O3	1.4411 (16)
C15—O2	1.1948 (15)		
C3—C2—C1	120.92 (12)	C12—C13—C16	117.80 (10)
C6—C7—C8	119.59 (11)	C5—O1—C9	118.03 (9)
C12—C11—C15	117.85 (10)		
O2—C15—O3—C17	−3.2 (2)	O4—C16—O5—C18	0.9 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···O4^{i}	0.93	2.53	3.4117 (15)	159
C17—H17B···O2^{ii}	0.96	2.60	3.557 (2)	178

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

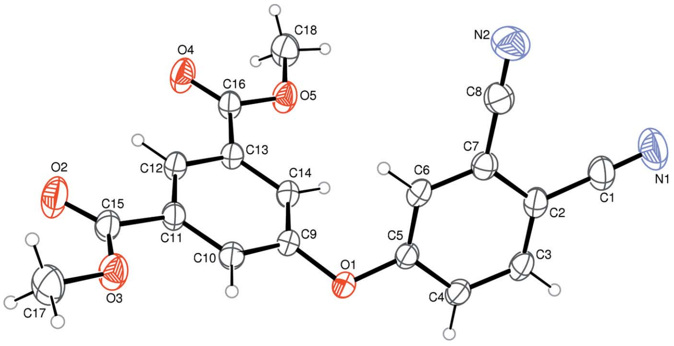


Figure 1
A view of the molecular structure of (I), with the atom-numbering scheme and 30% probability displacement ellipsoids.

H atoms were located geometrically and refined using a riding model, fixing the aromatic C—H distances at 0.93 Å and methyl C—H distances at 0.96 Å. U_{iso}(H) values were set to 1.5U_{eq}(methyl group) and 1.2U_{eq}(other carrier atoms).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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o2112 Petek *et al.* • $C_{18}H_{12}N_2O_5$ *Acta Cryst.* (2006). E**62**, o2111–o2112