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4-(3,5-Dimethoxyphenoxy)phthalonitrile

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.038
wR factor = 0.111
Data-to-parameter ratio = 19.0

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

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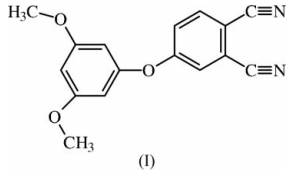
The crystal structure of the title compound, $C_{16}H_{12}N_2O_3$, is composed of a 3,5-dimethoxyphenoxy group attached to a phthalonitrile group. The structure is stabilized by intermolecular C—H···N contacts, in additional to weak van der Waals interactions.

Comment

Monosubstituted phthalonitriles, such as 4-(3,5-dimethoxyphenoxy)phthalonitrile, (I), are generally used for the synthesis of symmetrically and unsymmetrically substituted phthalocyanines and subphthalocyanines (Leznoff & Lever, 1989–1996). Phthalocyanines have continuously been the subject of research due to their wide-ranging applications, such as in organic pigments, chemical sensors, electrochromic display devices, photovoltaic cells, xerography, optical disks, catalysis and non-linear optics (McKeown, 1998).



(I)

Fig. 1 shows a perspective view of the molecule of (I), with the atom-numbering scheme. The triple-bond distance in the cyano groups is in good agreement with our previous reports (Köysal *et al.*, 2003; Köysal *et al.*, 2004; Ocak *et al.*, 2003).

Atoms C9/C10/C11/C12/C13/C14 are coplanar, with a maximum deviation of 0.17 (14) Å for atom C12; atoms O2 and O3 are 0.0022 (12) and 0.0082 (10) Å, respectively, from this plane. The dihedral angle between the C9–C14 and C1–C6 rings is 59.33 (4)°.

The crystal structure has an intermolecular C—H···N hydrogen bond, C2—H2···N2^{i} [symmetry code: (i) x, $-y-\frac{1}{2}$,

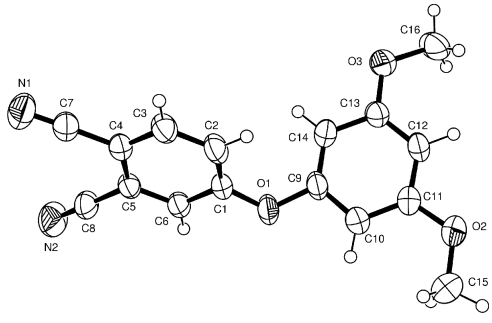


Figure 1
The structure of the title compound, (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

$\frac{1}{2}+z$]. In the crystal packing, the 3,5-dimethoxyphenoxy groups are close to each other, with van der Waals interactions, and are stacked parallel to the *c* axis of the unit cell.

Experimental

3,5-Dimethoxyphenol (1.08 g, 7 mmol) and 4-nitrophthalonitrile (1.00 g, 5.78 mmol) were dissolved in dry DMF (40 ml) with stirring under N_2. Dry fine-powdered sodium carbonate (1.0 g, 7.24 mmol) was added in portions (10 × 1 mmol) every 10 min. The reaction mixture was stirred for 48 h at room temperature and poured into iced water (150 g). The product was filtered off and washed with (10% *w*/*w*) NaOH solution and water until the filtrate was neutral. Recrystallization from ethanol gave a white product (yield 1.20 g, 74.07%). Single crystals were obtained from absolute ethanol at room temperature *via* slow evaporation (m.p. 413 K). Analysis calculated for $C_{16}H_{12}N_2O_3$: C 68.57, H 4.32, N 10.00%; found: C 68.46, H 4.40, N 9.90%. IR data (ν_{max}/cm^{-1}): 3056–3022 (Ar-CH_2), 2960–2856 (CH_2), 2229 (CN).

Crystal data

$C_{16}H_{12}N_2O_3$	D_x = 1.322 Mg m^{-3}
M_r = 280.28	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 9537 reflections
a = 12.8923 (12) Å	θ = 1.4–28.6°
b = 7.4816 (5) Å	μ = 0.09 mm^{-1}
c = 16.2916 (18) Å	T = 293 (2) K
β = 116.331 (7)°	Prism, colourless
V = 1408.4 (2) Å^3	0.50 × 0.34 × 0.25 mm
Z = 4	

Data collection

Stoe IPDS-2 diffractometer	R_{int} = 0.060
ω scans	θ_{max} = 28.8°
Absorption correction: none	$h = -17 \rightarrow 17$
17 632 measured reflections	$k = -10 \rightarrow 9$
3632 independent reflections	$l = -22 \rightarrow 21$
2238 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.062P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.111	$\Delta\rho_{max}$ = 0.18 e Å^{-3}
S = 0.97	$\Delta\rho_{min}$ = −0.15 e Å^{-3}
3632 reflections	Extinction correction: *SHELXL*97
191 parameters	Extinction coefficient: 0.021 (2)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C7—N1	1.1347 (16)	C13—O3	1.3621 (15)
C8—N2	1.1328 (17)	C15—O2	1.4196 (18)
C9—O1	1.3985 (13)	C16—O3	1.4304 (16)
C11—O2	1.3601 (15)		
C1—O1—C9	121.84 (9)	C13—O3—C16	116.67 (10)
C11—O2—C15	117.62 (11)		
C10—C11—O2—C15	3.9 (2)	C12—C13—O3—C16	14.54 (18)
C12—C11—O2—C15	−175.83 (13)	C14—C13—O3—C16	−164.74 (12)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2···N2^{i}	0.93	2.51	3.2621 (18)	138

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

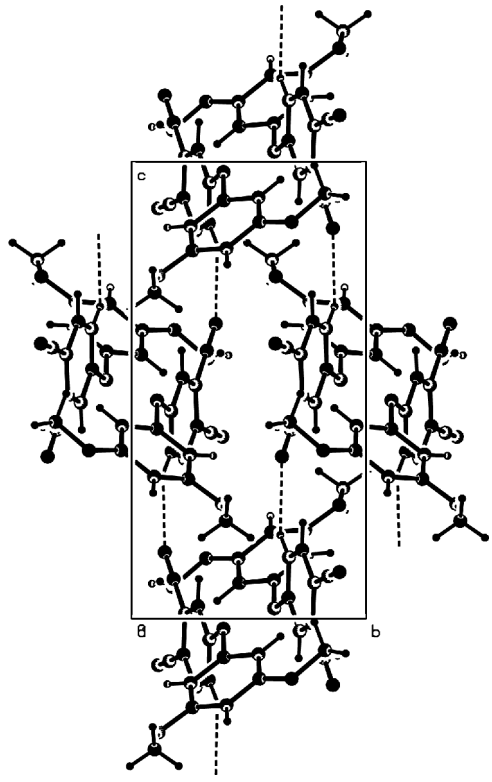


Figure 2
The crystal packing of the title compound. Dashed lines indicate hydrogen bonds.

H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C—H distances at 0.93 Å and the methyl C—H distances at 0.96 Å. The U_{iso}(H) values were set at 1.2U_{eq}(C) [1.5U_{eq}(C) for methyl atoms].

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

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