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# 4-(2-Methoxy-4-methylphenoxy)phthalonitrile

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**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean σ(C–C) = 0.002 Å
*R* factor = 0.043
*wR* factor = 0.117
Data-to-parameter ratio = 14.8

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

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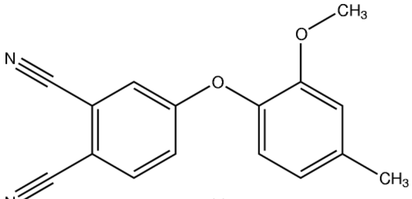
In the title compound, $C_{16}H_{12}N_2O_2$, the dihedral angle between the two benzene rings is 85.53 (5)°. The crystal structure is stabilized by intermolecular C—H···O hydrogen bonds and C—H···π interactions.

## Comment

4-(2-Methoxy-4-methylphenoxy)phthalonitrile, (I), is a starting material in the synthesis of peripherally tetrasubstituted phthalocyanines (Leznoff & Lever, 1989–1996). Phthalocyanines are an interesting class of compounds, with increasingly diverse industrial and biomedical applications, including photosensitization, linear optics, catalysis, liquid crystals and gas sensing (McKeown, 1998; Leznoff & Lever, 1989–1996).



(I)

The molecular structure of (I) is shown in Fig. 1. The dihedral angle between the two benzene rings is 85.53 (5)°. The C≡N bond lengths, both 1.140 (2) Å, agree with literature values (Ocak *et al.*, 2003). The geometry around the O atoms is in good agreement with the literature (Atalay *et al.*, 2003, 2004; Köysal *et al.*, 2004). Details of bond distances and angles are listed in Table 1.

The crystal structure of (I) is stabilized by an intermolecular C—H···O hydrogen bond and a weak C—H···π interaction (Table 2).

## Experimental

2-Methoxy-4-methylphenol (1.19 g, 8.61 mmol) and 4-nitrophthalonitrile (1.00 g, 5.78 mmol) were dissolved in dry dimethylformamide (DMF, 40 ml), with stirring, under $N_2$. Dry finely powdered potassium carbonate (1.2 g, 8.69 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 ice-water (150 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 1.52 g (87.58%). Single crystals were obtained by slow evaporation of an absolute ethanol solution at room temperature (m.p. 370 K). Analysis calculated for $C_{16}H_{12}N_2O_2$: C 72.72, H 4.58, N 10.60%; found: C 72.62, H 4.62, N 10.56%. $^1$H NMR ($CDCl_3$, p.p.m.): 2.41 (*s*, 3H, $CH_3$), 3.76 (*s*, 3H, $OCH_3$), 6.83–7.73 (*m*, 7H, Ar); $^{13}$C NMR ($CDCl_3$, p.p.m.): 21.44 ($CH_3$), 55.68 ($OCH_3$),

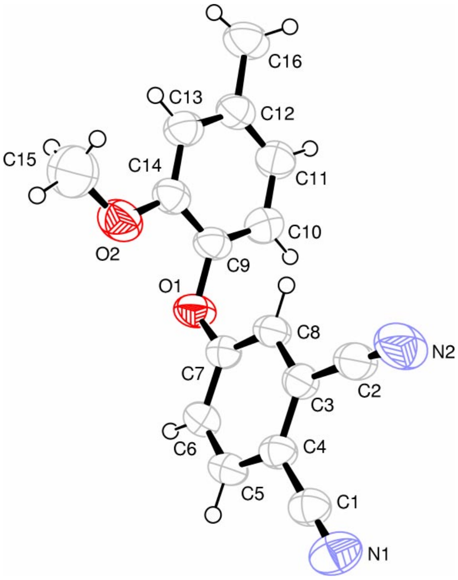


**Figure 1**

A view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids.

108.10, 113.90, 115.18, 115.58, 117.27, 120.28, 120.39, 121.94, 122.12, 135.08, 137.80, 139.00, 150.80, 162.01.

*Crystal data*

$C_{16}H_{12}N_2O_2$
$M_r = 264.28$
Orthorhombic, *Pbca*
$a = 8.2319$ (5) Å
$b = 22.7570$ (15) Å
$c = 14.6764$ (8) Å
$V = 2749.4$ (3) Å$^3$
$Z = 8$
$D_x = 1.277$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 18 160 reflections
$\theta = 1.4$–27.2°
$\mu = 0.09$ mm$^{-1}$
$T = 293$ (2) K
Plate, colorless
0.80 × 0.48 × 0.12 mm

*Data collection*

Stoe IPDS-II diffractometer
$\omega$ scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
$T_{\min} = 0.943$, $T_{\max} = 0.990$
18 348 measured reflections
2697 independent reflections
1818 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.110$
$\theta_{\max} = 26.0°$
$h = -10 \rightarrow 10$
$k = -28 \rightarrow 28$
$l = -16 \rightarrow 18$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.117$
$S = 0.91$
2697 reflections
182 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0722P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 0.18$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.15$ e Å$^{-3}$
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0125 (12)

**Table 1**

Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| C9—O1 | 1.3985 (17) | O2—C15 | 1.410 (3) |
| O1—C7 | 1.3595 (17) | C1—N1 | 1.140 (2) |
| C14—O2 | 1.3636 (19) | C2—N2 | 1.140 (2) |
| | | | |
| C7—O1—C9 | 119.87 (11) | C14—O2—C15 | 117.58 (15) |

**Table 2**

Hydrogen-bonding geometry (Å, °).

*Cg*2 is the centroid of the C9–C14 ring.

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C8—H8···O2$^{i}$ | 0.93 | 2.59 | 3.4276 (18) | 150 |
| C16—H16*C*···*Cg*2$^{i}$ | 0.96 | 2.99 | 3.6114 (18) | 124 |

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

All H atoms were positioned geometrically and refined using a riding model, with $C_{\rm aromatic}$—H = 0.93 Å, $C_{\rm methyl}$—H = 0.96 Å and $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

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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