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4-(2-Isopropyl-5-methylphenoxy)phthalonitrile

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.062
wR factor = 0.219
Data-to-parameter ratio = 21.8

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

In the title compound, $C_{18}H_{16}N_2O$, two benzene rings are connected by an O atom, and the dihedral angle between them is 86.83 (1)°. The C—O—C angle is 118.50 (14)°.

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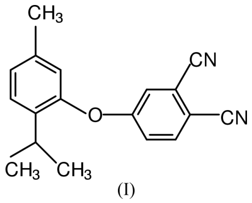
Comment

The title compound, (I), is a precursor in the synthesis of tetrasubstituted phthalocyanines (Leznoff & Lever, 1996). Substituted and unsubstituted phthalocyanines are widely used as pigments and dyes (Maser & Thomas, 1983). The production of phthalocyanines for the use of dyes and pigments is around 80,000 tons per year (Wöhrle, 2001).



(I)

The molecular structure of (I) is shown in Fig. 1 and a packing view is shown in Fig. 2. The N1≡C8 and N2≡C7 bond distances of 1.134 (2) and 1.144 (3) Å (Table 1), respectively, correspond to literature values (Çoruh *et al.*, 2002; Pandi *et al.*, 2001). The C—C bond distances and C—C—C angles in the two benzene rings are in good agreement with the expected values for aromatic rings.

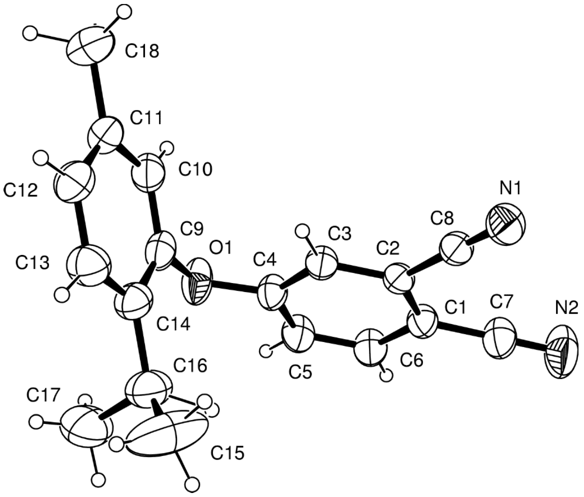


Figure 1
The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level.

Experimental

2-Isopropyl-5-methylphenol (1.94 g, 12.92 mmol) was dissolved in anhydrous dimethylformamide (50 ml) and 4-nitrophthalonitrile (1.90 g, 10.99 mmol) was added. After stirring for 20 min at room temperature, dry fine-powdered potassium carbonate (2.76 g, 20 mmol) was added portionwise over a period of 2 h with vigorous stirring. The reaction was stirred at room temperature for 24 h. Ice–water (200 g) was then added and the product filtered off and washed with water until the filtrate was neutral. The crude product was dissolved in 100 ml $CHCl_3$ and filtered. The $CHCl_3$ solution was then washed with 100 ml 10% Na_2CO_3 solution followed by distilled water. After drying the $CHCl_3$ solution over dry Na_2SO_4, the solvent was evaporated and the nitrile was obtained as a viscous oil. Recrystallization from *n*-heptane gave a brown product which was soluble in methanol, ethanol, *n*-heptane, $CHCl_3$ and $CHCl_2$. Single crystals of (I) were obtained from ethanol at room temperature *via* slow evaporation. M.p.: 383–385 K. IR(ν_{max}/cm^{-1}): 3070–3020 (Ar—CH), 2940–2840 (CH), 2220 (CN), 1616, 15, 85, 1560, 1500, 1472, 1448, 1404, 1380, 1368, 1340, 1308, 1276, 1196, 1160, 1112, 1080, 1060, 1000, 955, 928, 872, 845, 815, 764, 748, 724, 690, 615, 560, 525. ^{1}H NMR ($CDCl_3$): δ 1.17 (*d*, 6H), 2.33 (*s*, 3H), 2.96 (*m*, 1H), 6.74–7.72 (*m*, 6H); ^{13}C NMR ($CDCl_3$): δ 20.68, 22.93, 26.79, 106.21, 114.94, 115.36, 117.48, 120.71, 120.74, 121.25, 127.57, 135.32, 137.38, 150.23, 162.15. Analysis calculated for $C_{18}H_{16}N_2O$: C 78.24, H 5.84, N 10.14%; found C 78.20, H 5.56, N 10.10%. Yield: 1.34 g (44.2%).

Crystal data

$C_{18}H_{16}N_2O$
$M_r = 276.33$
Monoclinic, $P2_1/n$
$a = 11.424 (2)$ Å
$b = 10.2230 (12)$ Å
$c = 13.669 (2)$ Å
$\beta = 102.406 (1)^\circ$
$V = 1559.1 (4)$ Å^3
$Z = 4$

$D_x = 1.177$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 15187 reflections
$\theta = 1.8–29.4^\circ$
$\mu = 0.07$ mm^{-1}
$T = 293 (2)$ K
Prism, light brown
$0.40 \times 0.33 \times 0.30$ mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: none
14570 measured reflections
4223 independent reflections
2185 reflections with $I > 2\sigma(I)$

$R_{int} = 0.037$
$\theta_{max} = 29.5^\circ$
$h = -15 \rightarrow 15$
$k = -13 \rightarrow 14$
$l = -16 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.219$
$S = 0.88$
4223 reflections
194 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1493P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.43$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N2—C7	1.144 (3)	C16—C14	1.514 (3)
N1—C8	1.134 (2)	C4—O1	1.363 (2)
C7—C1	1.429 (3)	C9—O1	1.413 (2)
C8—C2	1.437 (2)	C11—C18	1.507 (3)
N2—C7—C1	179.7 (3)	O1—C4—C3	123.64 (16)
N1—C8—C2	178.3 (2)	C10—C9—O1	117.62 (19)
C9—C14—C16	121.2 (2)	C4—O1—C9	118.50 (14)

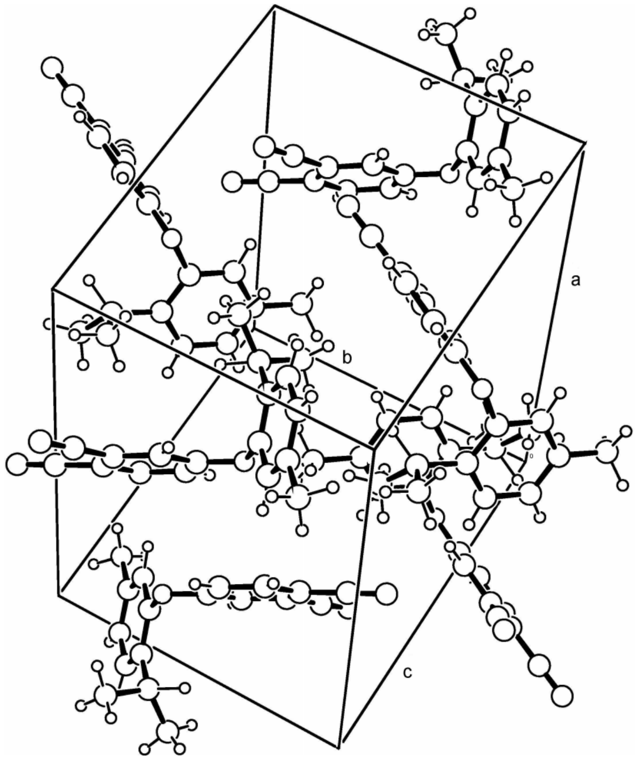


Figure 2
A packing diagram of (I), showing part of the stacking of molecules.

The H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C—H distance at 0.93 Å, the methyl C—H distance 0.96 Å.

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, 1997) and *PARST* (Nardelli, 1995).

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o1112 Şehriman Atalay *et al.* • $C_{18}H_{16}N_2O$ *Acta Cryst.* (2003). E**59**, o1111–o1112