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Key indicators

Single-crystal X-ray study
 $T = 295$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
Disorder in main residue
 R factor = 0.052
 wR factor = 0.113
Data-to-parameter ratio = 11.9

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

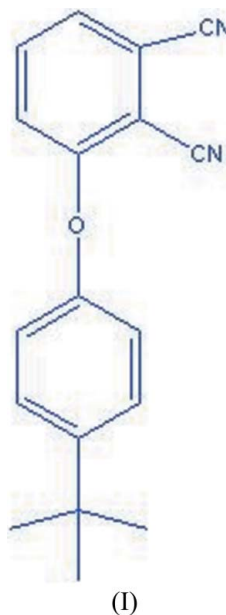
3-(4-*tert*-Butylphenoxy)phthalonitrile

The title compound, $\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}$, contains a phthalonitrile ring lying on a crystallographic mirror plane and a 4-*tert*-butylphenoxy substituent oriented exactly perpendicular to that plane.

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Comment

Substituted phthalonitriles are generally used for preparing symmetrically and unsymmetrically peripherally substituted phthalocyanine complexes and sub-phthalocyanines (McKeown, 1998; Leznoff & Lever, 1989–1996). Phthalocyanines were first developed as dyes and pigments (Moser & Thomas, 1983). Over the last few years, a great deal of interest has focused on the synthesis of phthalocyanine derivatives due to their applications in many fields, such as chemical sensors, electrochromic devices, batteries, semiconductive materials, liquid crystals, non-linear optics and photodynamic therapy (PDT) (Leznoff & Lever, 1989–1996).



We report here the structure of the title compound, (I), in which the phthalonitrile ring lies on a crystallographic mirror plane with atoms C10 and C11 of the phenoxy ring and atom C14 of the disordered *tert*-butyl substituent in general positions (Fig. 1). As a result, the 4-*tert*-butylphenoxy ring plane is oriented exactly perpendicular to that of the phthalocyanine ring. The $\text{C1}\equiv\text{N1}$ and $\text{C2}\equiv\text{N2}$ bond distances are 1.137 (6) and 1.139 (6) Å, respectively, consistent with $\text{N}\equiv\text{C}$ triple-bond character, and in good agreement with literature values (Nesi *et al.*, 1998; Dinçer *et al.*, 2004; Ocak *et al.*, 2004).

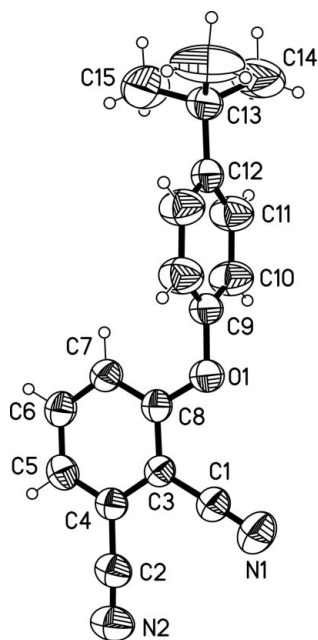


Figure 1
The structure of the title compound, (I), showing 35% probability ellipsoids and the atomic numbering scheme. The C and H atoms of the methyl groups of the minor disorder component (C14' and C15') have been omitted for clarity. Unlabeled atoms are related to labeled atoms by $x, -y + \frac{3}{2}, z$.

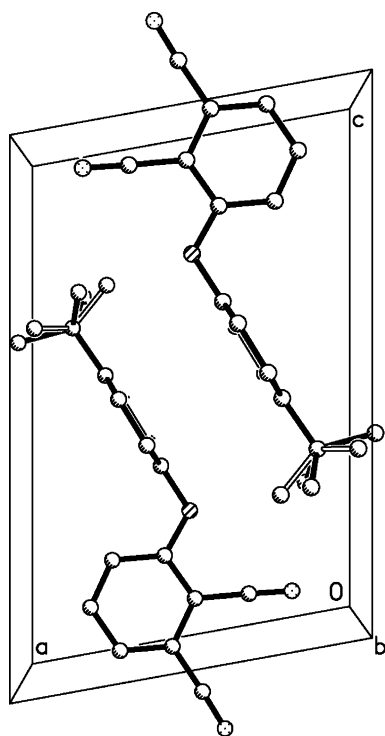


Figure 2
The molecular packing of (I), viewed along the b axis. H atoms have been omitted.

Experimental

4-*tert*-Butylphenol (3 g, 20 mmol) and 3-nitrophthalonitrile (1.73 g, 10 mmol) were dissolved in dry dimethylformamide (50 ml). After

stirring for 1 h at room temperature, dry finely powdered potassium carbonate (2.76 g, 20 mmol) was added portionwise over a period of 2 h with stirring. The reaction mixture was stirred for 36 h at room temperature and poured into ice–water (200 g). The product was filtered off and washed with water until the filtrate was neutral. Recrystallization from toluene gave a white product (yield 1.97 g, 71.4%). Single crystals were obtained from toluene at room temperature by slow evaporation (m.p. 393–394 K). IR (KBr, ν cm^{-1}): 2964, 2868, 2239, 2229; ^1H NMR (CDCl_3): δ 7.54–7.41 (m , 4H), 7.10–7.00 (m , 3H), 1.34 (s , 9H); ^{13}C NMR (CDCl_3): δ 161.4, 151.4, 149.4, 134.4, 129.1, 127.4, 126.8, 125.4, 120.4, 120.0, 117.3, 115.3, 112.9, 105.9, 34.7, 31.5.

Crystal data

$\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}$
 $M_r = 276.33$
Monoclinic, $P2_1/m$
 $a = 8.7541$ (10) Å
 $b = 6.8132$ (5) Å
 $c = 13.5203$ (19) Å
 $\beta = 100.280$ (13)°
 $V = 793.45$ (16) Å³
 $Z = 2$

$D_x = 1.157$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 38 reflections
 $\theta = 5.1$ – 12.4 °
 $\mu = 0.07$ mm⁻¹
 $T = 295$ (2) K
Prism, colorless
 $0.4 \times 0.3 \times 0.1$ mm

Data collection

Bruker P4 diffractometer
 ω scans
Absorption correction: none
2265 measured reflections
1621 independent reflections
757 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.032$

$\theta_{\text{max}} = 25.5$ °
 $h = -1 \rightarrow 10$
 $k = -1 \rightarrow 8$
 $l = -16 \rightarrow 16$
3 standard reflections
every 97 reflections
intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.052$
 $wR(F^2) = 0.113$
 $S = 1.01$
1621 reflections
136 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.001P)^2 + 0.3P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.006$
 $\Delta\rho_{\text{max}} = 0.16$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.10$ e Å⁻³

The methyl groups of the *tert*-butyl substituent on the phenoxy ring are disordered with respect to rotation about the C12–C13 bond. The two main orientations of the methyl groups were assigned as C14/C15 and C14'/C15', and their occupancies refined to 0.670 (3) and 0.330 (3), respectively. H atoms, including those on the disordered methyl C atoms, were positioned 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 [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{aromatic C})$ or $U_{\text{eq}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl C})$].

Data collection: XSCANS (Bruker, 1997); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structure: SHELXTL (Bruker, 1997); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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