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4-(4-Benzyloxyphenoxy)phthalonitrile

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The title compound, $C_{21}H_{14}O_2N_2$, consists of two substituted benzene rings and one phthalonitrile moiety. The dihedral angle between the first two benzene rings is 62.91 (1)°, and that between the phthalonitrile and the central benzene ring is 70.82 (1)°.

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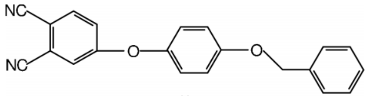
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.056
wR factor = 0.179
Data-to-parameter ratio = 13.9

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

Comment

Monosubstituted phthalonitriles have been used as starting materials for symmetrically and unsymmetrically monosubstituted phthalocyanines and subphthalocyanines (McKeown, 1998), which are important components for dyes, pigments, gas sensors, optical limiters and liquid crystals, and which are also used in medicine, as singlet oxygen photosensitizers for photodynamic therapy (PDT; Leznoff & Lever, 1996). The production of phthalocyanines for the use of dyes and pigments is around 80000 tons per year (Wöhrle, 2001). We report here the structure of the title compound, (I) (Fig. 1).



(I)

All the C—C bond distances in the benzene rings have typical Csp^2—Csp^2 values. The average C—C bond distances in rings C7–C12 and C14–C19 are 1.362 (5) and 1.402 (4) Å. The C4—O1 and C7—O1 bond distances are 1.373 (4) and 1.405 (4) Å, which are similar observed values in the literature (Gales *et al.*, 2001; Tesouro Vallina & Stoeckli-Evans, 2001; Karadayı *et al.*, 2003). The C20≡N1 and C21≡N2 bond distances are 1.142 (5) and 1.154 (5) Å, respectively. These values are within the expected ranges for phthalonitrile derivatives (Işik *et al.*, 1999; Öztürk *et al.*, 1999). The three benzene rings are each planar within experimental error, the largest deviation being 0.0119 (2) Å for atom C5 of the C1–C6 ring. The dihedral angle between the rings C1–C6 and C7–C12

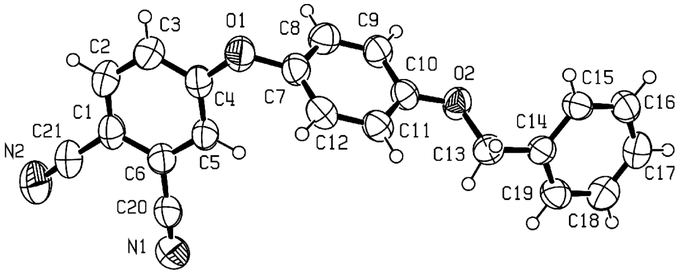


Figure 1
The molecular structure of (I) and the crystallographic numbering scheme adopted. Displacement ellipsoids are drawn at the 50% probability level.

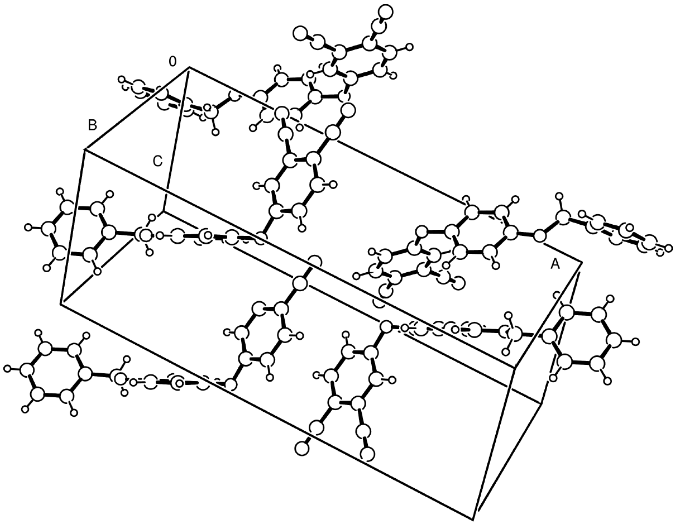


Figure 2
The crystal structure of (I).

is 71.31 (1)°, that between C7–C12 and C14–C19 is 62.91 (1)°, and that between C1–C6 and C14–C19 is 37.66 (1)°. The crystal structure of (I) is shown in Fig. 2.

Experimental

4-Benzyloxyphenol (1.20 g, 6 mmol) and 4-nitrophtalonitrile (1.0 g, 5.78 mmol) were heated at 333 K in dry DMF (35 ml), with stirring, under N_2. Dry fine-powdered potassium carbonate (0.96 g, 6.96 mmol) was added portionwise over 2 h with stirring. The solution was stirred for 48 h at 333 K and poured into ice–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. Crystals of (I) were obtained from ethanol at room temperature *via* slow evaporation (yield 60%; m.p. 383 K). Elemental analysis calculated for $C_{21}H_{14}N_2O_2$: C 77.27, H 4.32, N 8.58%; found: C 77.50, H 4.10, N 8.50%. IR (KBr, ν, cm^{-1}): 3107–3037 (Ar-CH_2), 2870–2940 (CH_2), 2229 (CN), 1603, 1587, 1502, 1485, 1412, 1381, 1306, 1298, 1238, 1200, 1084, 1013, 949, 852, 841, 827, 748, 696, 633.

Crystal data

$C_{21}H_{14}N_2O_2$
$M_r = 326.36$
Monoclinic, $P2_1/c$
$a = 20.149$ (3) Å
$b = 10.8606$ (8) Å
$c = 7.6424$ (10) Å
$\beta = 81.892$ (11)°
$V = 1655.7$ (4) Å^3
$Z = 4$
$D_x = 1.309$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4810 reflections
$\theta = 1.9$–25.0°
$\mu = 0.09$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.42 × 0.31 × 0.10 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: none
11417 measured reflections
3163 independent reflections
1505 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.071$
$\theta_{\max} = 26.0°$
$h = -24 \rightarrow 24$
$k = -13 \rightarrow 12$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.057$
$wR(F^2) = 0.179$
$S = 0.99$
3163 reflections
227 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0862P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.004$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.019 (3)

Table 1
Selected geometric parameters (Å, °).

N1—C20	1.142 (5)	O2—C13	1.432 (4)
C20—C6	1.438 (5)	O1—C4	1.373 (4)
C21—N2	1.154 (5)	O1—C7	1.405 (4)
C21—C1	1.427 (5)	C14—C13	1.492 (4)
O2—C10	1.366 (4)		
N1—C20—C6	178.9 (4)	C15—C14—C13	119.7 (3)
N2—C21—C1	177.8 (5)	C12—C7—O1	121.7 (3)
C10—O2—C13	118.7 (2)	C8—C7—O1	117.3 (3)
C4—O1—C7	118.3 (3)	O2—C13—C14	107.5 (2)
O2—C10—C9	114.7 (3)	O1—C4—C5	123.7 (3)
O2—C10—C11	125.6 (3)	O1—C4—C3	115.6 (3)
C19—C14—C13	121.5 (3)		
C13—O2—C10—C9	−177.3 (3)	C10—O2—C13—C14	178.1 (3)
C4—O1—C7—C8	95.4 (4)	C15—C14—C13—O2	61.5 (4)

The H atoms were placed in calculated positions and refined using a riding model, with C—H distances of 0.93 Å for Csp^2—H bonds and 0.97 Å for methylene C—H, and $U_{\rm iso}$(H) values were set at $1.2U_{\rm eq}$(parent atom).

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) and *PLUTON* (Spek, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

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