Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

4-(4-Heptyloxyphenoxy)phthalonitrile

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.118
Data-to-parameter ratio = 16.2

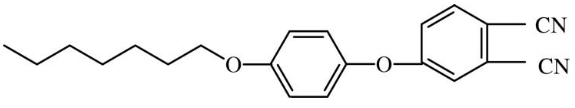
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 3 January 2006
Accepted 16 January 2006

In the crystal structure of the title compound, $C_{21}H_{22}N_2O_2$, the 4-heptyloxyphenoxy group and phthalonitrile group are both planar. The dihedral angle between the two benzene rings is 86.30 (6)°

Comment

4-Substituted phthalonitriles are generally used for preparing symmetrically and unsymmetrically peripherally substituted phthalocyanine complexes and subphthalocyanines (McKeown, 1998; Leznoff & Lever, 1989–1996). Phthalocyanines are traditionally used as dyes and pigments (Moser & Thomas, 1983). Other areas of current interest include applications in catalysis, chemical sensors, electrochromism, batteries, photodynamic therapy, semiconductive materials, liquid crystals and modified supports for gas-solid chromatography (Leznoff & Lever, 1989–1996).



(I)

The molecular structure of the title compound, (I), is shown in Fig. 1. The C≡N triple-bond distances in the molecule are in good agreement with those in previously published structures (Ocak *et al.*, 2003; Köysal *et al.*, 2003, 2004).

Atoms C9–C14 are coplanar, the largest deviation being 0.013 (1) Å at C12. Atoms O1 and O2 deviate from this plane by 0.138 (3) and 0.047 (3) Å, respectively. The dihedral angle between the C9–C14 and C1–C6 rings is 86.30 (6)°.

The displacement parameters of atoms C15–C21 of the heptyloxy group are larger than those for other atoms. The high values of the anisotropic displacement parameters associated with the heptyl group suggest that this group could be affected by high thermal motion.

Experimental

4-Heptyloxyphenol (1.40 g, 67.21 mmol) and 4-nitrophthalonitrile (1.10 g, 63.58 mmol) were dissolved in dry dimethylformamide (DMF, 40 ml) with stirring under N_2. Dry fine-powdered potassium carbonate (1.5 g, 10.87 mmol) was added in portions (10 × 1 mmol) every 10 min. The reaction mixture was stirred for 48 h at 323 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 (yield 1.56 g, 73.38%). Single crystals were obtained from dry DMF at room temperature by slow evaporation (m.p. 330–333 K).

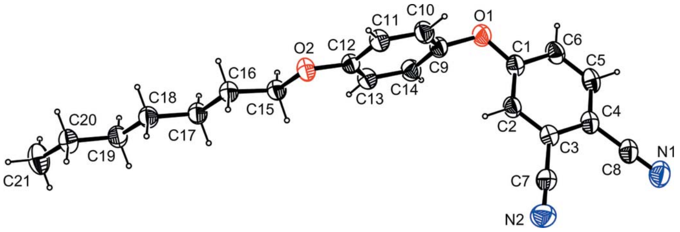


Figure 1
The structure of the title compound, (I), with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

Crystal data

$C_{21}H_{22}N_2O_2$
M_r = 334.41
Triclinic, $P\overline{1}$
a = 8.4862 (15) Å
b = 8.6420 (14) Å
c = 14.727 (3) Å
α = 91.261 (14)°
β = 98.442 (14)°
γ = 118.186 (12)°
V = 936.5 (3) Å^3
Z = 2
D_x = 1.186 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 9434 reflections
θ = 2.7–28.0°
μ = 0.08 mm^{-1}
T = 293 (2) K
Prism, colourless
0.39 × 0.22 × 0.08 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{\min}$ = 0.971, $T_{\max}$ = 0.994
12093 measured reflections
3686 independent reflections
2011 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.084
$\theta_{\max}$ = 26.0°
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 9$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.118
S = 0.97
3686 reflections
227 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.13 e Å^{-3}
$\Delta\rho_{\min}$ = −0.12 e Å^{-3}

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 (aromatic), 0.97 (methylene) and 0.96 Å (methyl), with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(aromatic and methylene) and 1.5$U_{\rm eq}$(methyl).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

Thanks are expressed to Nesuhi Akdemir and Erbil Ãgar for their assistance.

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