

1,3-Bis(3,4-dicyanophenoxy)benzene

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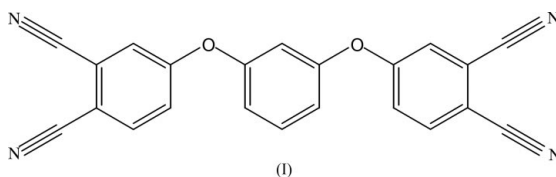
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Key indicators

Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
R factor = 0.065
wR factor = 0.186
Data-to-parameter ratio = 17.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.The title compound, $\text{C}_{22}\text{H}_{10}\text{N}_4\text{O}_2$, has been designed and synthesized as a precursor of dye pigments.Received 28 October 2004
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Comment

The title compound, (I), is a precursor in the synthesis of network polymeric phthalocyanines and high-performance aromatic polymers (McKeown, 1998; Takekoshi, 1987). Phthalocyanines are traditionally used as dyes and pigments (Moser & Thomas, 1983). They belong to an interesting class of compounds with increasingly diverse industrial and biomedical applications, including photosensitization, linear optics, catalysis, liquid crystals and gas sensing (Leznoff & Lever, 1989–1996).

The molecular structure of (I) is shown in Fig. 1. Selected bond lengths and angles are given in Table 1. Compound (I) has four cyano groups. The $\text{C}7\equiv\text{N}1$, $\text{C}8\equiv\text{N}2$, $\text{C}21\equiv\text{N}3$ and $\text{C}22\equiv\text{N}4$ bond distances are 1.137 (2), 1.144 (2), 1.140 (2) and 1.138 (2) Å , respectively, showing $\text{N}\equiv\text{C}$ triple-bond character. These values agree well with literature values (Ocak *et al.*, 2004).

Experimental

1,3-Benzenediol (0.32 g, 2.91 mmol) and 4-nitrophthalonitrile (1.00 g, 5.78 mmol) were dissolved in dry dimethylformamide (40 ml) with stirring under N_2 . Dry fine-powdered potassium carbonate (1.14 g, 8.26 mmol) was added in portions ($10 \times 1 \text{ mmol}$) 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 an NaOH solution (10%) and water until the filtrate was neutral. Recrystallization from ethanol gave a brown product (yield 0.55 g, 52.43%). Single crystals were obtained from ethanol at room temperature *via* slow evaporation (m.p. 458 K); elemental analysis calculated for $\text{C}_{22}\text{H}_{10}\text{N}_4\text{O}_2$: C 72.93, H 2.78, N 15.46%; found: C 72.83, H 2.80, N 15.56%. IR: 3090–3038 (Ar-CH₂), 2227 (CN).

Crystal data

 $\text{C}_{22}\text{H}_{10}\text{N}_4\text{O}_2$
 $M_r = 362.34$
Triclinic, $P\bar{1}$
 $a = 8.2296 (6) \text{ \AA}$
 $b = 8.6309 (7) \text{ \AA}$
 $c = 13.4708 (10) \text{ \AA}$
 $\alpha = 75.085 (6)^\circ$
 $\beta = 88.710 (6)^\circ$
 $\gamma = 77.777 (6)^\circ$
 $V = 903.10 (12) \text{ \AA}^3$ Z = 2
 $D_x = 1.333 \text{ Mg m}^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 16095 reflections
 $\theta = 1.6\text{--}28.4^\circ$
 $\mu = 0.09 \text{ mm}^{-1}$
T = 293 (2) K
Prism., brown
 $0.80 \times 0.42 \times 0.10 \text{ mm}$

Data collection

Stoe IPDS-2 diffractometer
 ω scans
 Absorption correction:
 by integration (*X-RED32*;
 Stoe & Cie, 2002)
 $T_{\min} = 0.946$, $T_{\max} = 0.991$
 16274 measured reflections

4496 independent reflections
 2856 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.155$
 $\theta_{\text{max}} = 28.4^\circ$
 $h = -11 \rightarrow 10$
 $k = -11 \rightarrow 11$
 $l = -17 \rightarrow 17$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.065$
 $wR(F^2) = 0.186$
 $S = 0.96$
 4496 reflections
 254 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1113P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.27 \text{ e \AA}^{-3}$
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.134 (15)

Table 1

Selected geometric parameters (\AA , $^\circ$).

O2—C15	1.3617 (18)	C21—N3	1.140 (2)
O2—C11	1.4033 (18)	C8—N2	1.144 (2)
C18—C21	1.435 (2)	C8—C2	1.437 (2)
C19—C22	1.440 (2)	C22—N4	1.138 (2)
O1—C5	1.372 (2)	C1—C7	1.436 (2)
O1—C9	1.382 (2)	C7—N1	1.137 (2)
C16—C15—C20	121.10 (14)	C11—C12—C13	118.26 (18)
C12—C11—C10	121.88 (15)	C4—C5—C6	121.33 (16)
C14—C9—C10	121.17 (17)	C9—C14—C13	118.67 (16)
C4—C3—C2	120.98 (16)	C12—C13—C14	121.50 (18)
C11—C10—C9	118.51 (16)		
C11—O2—C15—C16	6.1 (2)	C9—O1—C5—C6	51.8 (3)

H atoms were positioned geometrically and refined using a riding model, with a C—H distance of 0.93 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s)

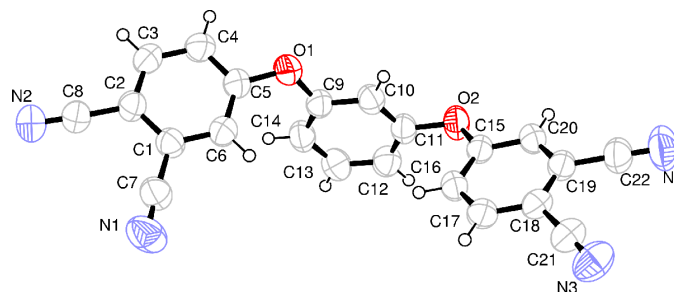


Figure 1

An *ORTEP*III (Burnett & Johnson, 1996) drawing of (I), showing the atomic numbering scheme. Displacements ellipsoids of non-H atoms are shown at the 50% probability level.

used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

- Burnett, M. N. & Johnson, C. K. (1996). *ORTEP*III. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Leznoff, C. C. & Lever, A. B. P. (1989–1996). *Phthalocyanines: Properties and Applications*, Vols. 1, 2, 3 & 4. Weinheim, New York: VHC Publishers Inc.
 McKeown, N. B. (1998). *Phthalocyanine Materials: Synthesis, Structure and Function*. Cambridge University Press.
 Moser, F. H. & Thomas, A. L. (1983). *The Phthalocyanines*, Vols. 1 and 2. Boca Raton, Florida: CRC Press.
 Ocak, N., Çoruh, U., Akdemir, N., Kantar, C., Açar, E. & Erdönmez, A. (2004). *Acta Cryst.* **E60**, o33–o34.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
 Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.
 Takekoshi, T. (1987). *Polym. J.* **19**, 191–202.