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Key indicators

Single-crystal X-ray study

T = 293 K

Mean $\sigma(\text{C}-\text{C}) = 0.005 \text{ \AA}$

R factor = 0.044

wR factor = 0.138

Data-to-parameter ratio = 11.7

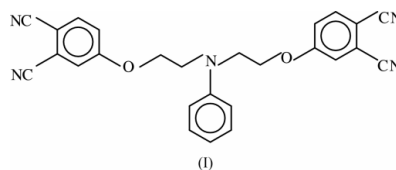
For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.4,4'-(*N*-Phenyl-2,2'-iminodiethoxy)-
diphthalonitrileThe title compound, $\text{C}_{26}\text{H}_{19}\text{N}_5\text{O}_2$, contains three aromatic
rings, which are not coplanar. The crystal structure is stabilized
by intermolecular $\text{C}-\text{H} \cdots \text{N}$ contacts.

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Comment

Diphthalonitriles have been used as starting materials for
network polymeric phthalocyanines (McKeown, 1998).
Phthalocyanine compounds have been widely studied for over
50 years due to their varied applications (Moser & Thomas,
1983). Polymeric phthalocyanines have been described for use
as dyes and industrial high-technology materials and are also
of additional interest because of their high thermostability
(Leznoff & Lever, 1989–1996).

An *ORTEP*III (Burnett & Johnson, 1996) plot of the title structure, (I), is shown in Fig. 1. The bond distances and angles in (I) are normal (Table 1). The average $\text{N}\equiv\text{C}$ bond distance in cyano groups, 1.139 (4) \AA , is short enough to indicate their triple-bond character. This value is in good agreement with those in 4,4'-[2,2-methylenebis(4-chlorophenoxy)]diphthalonitrile (Çoruh *et al.*, 2002) and 4-(phenothiazin-10-yl)benzene-1,2-dicarbonitrile (Öztürk *et al.*, 1999). The $\text{O}-\text{C}$ bond distances correspond to those in 4,4'-[2,2-methylenebis(4-chlorophenoxy)]diphthalonitrile (Çoruh *et al.*, 2002).

The three aromatic rings in the molecule are essentially planar. The dihedral angle between ring *A* (C13/C11/C4/C16/C15/C8) and ring *B* (C7/C21/C14/C17/C20/C12) is 70.73 (7)°, while the dihedral angle between ring *A* and ring *C* (C3/C18/C28/C29/C27/C5) is 73.54(0.10)°. The angle between rings *B* and *C* is 69.23(0.10)°.

The crystal structure is stabilized by intermolecular $\text{C}-\text{H} \cdots \text{N}$ contacts (Table 2).

Experimental

N-Phenyl-2,2'-iminodiethanol (1.17 g, 6.46 mmol) was dissolved in dry DMF and 4-nitrophthalonitrile (2.15 g, 12.43 mmol) was added. After stirring for 30 min, finely ground anhydrous K_2CO_3 (2.76 g, 20 mmol) was added portionwise over 2 h with vigorous stirring. The reaction mixture was stirred for 24 h at 333 K and then poured into ice water (200 g). The product was filtered off and washed with water until the filtrate was neutral. The product was then refluxed in

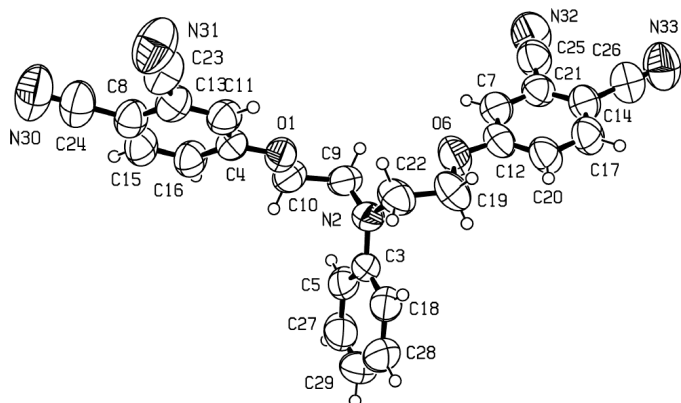


Figure 1
An ORTEPIII drawing (Burnett & Johnson, 1996) of the title compound, showing the atomic numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level.

methanol, filtered and dried. The title compound, (I), was crystallized from dimethylformamide by slow evaporation at room temperature (yield 0.29 g, 10.8%). M.p.: 433 K. IR ($\nu_{\max}/\text{cm}^{-1}$): 3100–3040 (Ar—CH), 2940–2900 (CH), 2220 (CN), 1660, 1588, 1556, 1484, 1452, 1424, 1388, 1360, 1288, 1240, 1192, 1168, 1116, 1092, 1028, 1016, 984, 952, 908, 888, 844, 828, 748, 725, 690, 640, 624, 590, 550, 520. ^1H NMR (acetone- d_6): 4.01 (*t*, 2H), 4.47 (*t*, 2H), 6.68–7.95 (*m*, 6H). ^{13}C NMR (acetone- d_6): 51.18 (CH₂—N), 68.17 (CH₂—O), 107.93, 113.52, 116.47, 117.06, 117.97, 120.75, 120.93, 130.19, 136.38, 147.94, 163.18. Analysis calculated for C₂₆H₁₉N₅O₂: C 72.04, H 4.42, N 16.16%; found: C 72.04, H 4.39, N 16.14%.

Crystal data

C ₂₆ H ₁₉ N ₅ O ₂	Cu K α radiation
$M_r = 433.46$	Cell parameters from 3683 reflections
Orthorhombic, <i>Pbca</i>	$\theta = 3.87$ – 64.5°
$a = 16.621$ (5) Å	$\mu = 0.65$ mm ⁻¹
$b = 9.004$ (5) Å	$T = 293$ (2) K
$c = 31.494$ (5) Å	Prism, dark yellow
$V = 4713$ (3) Å ³	0.25 × 0.18 × 0.13 mm
$Z = 8$	
$D_x = 1.222$ Mg m ⁻³	

Data collection

Nonius KappaCCD diffractometer	$R_{\text{int}} = 0.001$
ω – 2θ scans	$\theta_{\text{max}} = 63.0^\circ$
Absorption correction: none	$h = -18 \rightarrow 18$
6338 measured reflections	$k = -9 \rightarrow 9$
3484 independent reflections	$l = -36 \rightarrow 35$
1872 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0507P)^2 + 0.7824P]$
$R[F^2 > 2\sigma(F^2)] = 0.044$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.138$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.01$	$\Delta\rho_{\text{max}} = 0.11$ e Å ⁻³
3484 reflections	$\Delta\rho_{\text{min}} = -0.10$ e Å ⁻³
298 parameters	
H atoms treated by a mixture of independent and constrained refinement	

Table 1

Selected geometric parameters (Å, °).

O1—C4	1.344 (3)	O6—C19	1.436 (3)
O1—C10	1.437 (3)	C23—N31	1.135 (4)
N2—C3	1.378 (3)	C24—N30	1.144 (4)
N2—C9	1.443 (3)	C25—N32	1.137 (4)
N2—C22	1.445 (3)	C26—N33	1.139 (4)
O6—C12	1.352 (3)		
C4—O1—C10	118.2 (2)	C12—O6—C19	119.6 (2)
C3—N2—C9	121.2 (2)	N2—C9—C10	113.0 (2)
C3—N2—C22	121.7 (2)	N2—C22—C19	113.0 (2)
C9—N2—C22	117.1 (2)		

Table 2

Intermolecular contacts (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C19—H19A...N31 ⁱ	0.97	2.59	3.546 (5)	170

Symmetry code: (i) $x - \frac{1}{2}, \frac{3}{2} - y, 1 - z$.

The H atoms were located geometrically and refined using a riding model. The H atoms were located geometrically and refined using a riding model, fixing the aromatic C—H distance at 0.93 Å, the methylene C—H distance 0.97 Å.

Data collection: COLLECT (Nonius, 1997–2000); cell refinement: HKL SCALEPACK (Otwinowski & Minor, 1997); data reduction: HKL DENZO (Otwinowski & Minor, 1997) and SCALEPACK; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPIII (Burnett & Johnson, 1996); software used to prepare material for publication: WinGX (Farrugia, 1997) and PARST (Nardelli, 1995).

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