Acta Crystallographica Section E
**Structure Reports**
**Online**

ISSN 1600-5368

# Benzyl 2-(2-chloro-4,5-dicyanophenoxy)benzoate

**Nazan Ocak Ískeleli[a] and Ayşen Ağar[b]***

[a]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, TR-55139, Kurupelit-Samsun, Turkey, and [b]Department of Chemistry, Faculty of Arts and Sciences, Ondokuz Mayıs University, TR-55139, Kurupelit-Samsun, Turkey

Correspondence e-mail: nocak@omu.edu.tr

**Key indicators**

Single-crystal X-ray study
$T$ = 293 K
Mean $\sigma$(C–C) = 0.006 Å
$R$ factor = 0.037
$wR$ factor = 0.105
Data-to-parameter ratio = 10.8

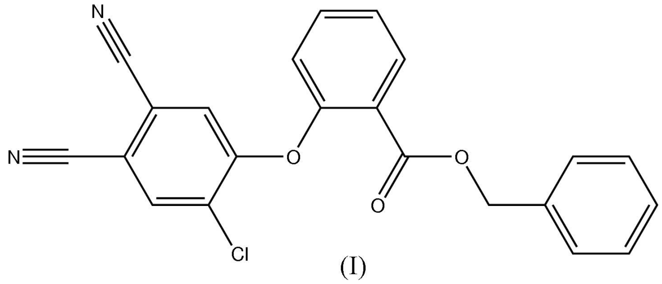
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title phthalonitrile derivative, $C_{22}H_{13}ClN_2O_3$, contains three aromatic rings, which are not coplanar. The crystal structure is stabilized by intermolecular C—H···N contacts.

Received 29 April 2005
Accepted 24 May 2005
Online 31 May 2005

## Comment

Phthalonitriles such as benzyl 2-(2-chloro-4,5-dicyanophenoxy)benzoate, (I), are generally used for the synthesis of symmetrically and unsymmetrically substituted phthalocyanines and subphthalocyanines (Leznoff & Lever, 1989–1996). Phthalocyanines have long been the subject of research as a result of their wide range of applications, such as in organic pigments, chemical sensors, electrochromic display devices, photovoltaic cells, xerography, optical disks, catalysis and nonlinear optics (McKeown, 1998).



(I)

The lengths of the two N≡C triple bonds in (I) are consistent with those found in similar compounds (Atalay *et al.*, 2003; Erdem *et al.*, 2004; Iskeleli & Ağar, 2005). The C═O bond distance is close to the values reported in dimethyl 2,2′-(4,5-dicyano-*o*-phenylenedioxy)dibenzoate (Ocak *et al.*, 2004). The dihedral angle between the planes of rings *A* (C3–C8) and *B* (C9–C14) is 72.56 (8)°, while the dihedral angle between the planes of rings *A* and *C* (C17–C22) is 3.16 (12)°. The angle between the planes of rings *B* and *C* is 69.96 (14)°.

The crystal structure of (I) is stabilized by C—H···N intermolecular contacts, namely C7—H7···N1$^{i}$ [symmetry code: (i) $-x, y+\frac{1}{2}, -z+1$].

## Experimental

Benzyl 2-hydroxybenzoate (3.54 g, 15.51 mmol) and 4-nitrophthalonitrile (1.0 g, 5.78 mmol) were dissolved in dry DMF (50 ml). After stirring for 30 min at 313 K, dry fine-powdered potassium carbonate (3.00 g, 21.74 mmol) was added portionwise over a period of 2 h with stirring. The reaction was stirred for 48 h at 313 K and poured into ice–water (200 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 1.78 g, 90.36%). Single crystals were obtained from absolute ethanol at room temperature *via* slow evaporation (m.p. 358 K). Analysis calculated for $C_{22}H_{13}ClN_2O_3$: C 67.96, H 3.37, N 7.20%; found: C 67.90, H 3.36, N 7.26%.

*Crystal data*

$C_{22}H_{13}ClN_2O_3$
$M_r = 388.79$
Monoclinic, $P2_1$
$a = 8.1240\ (10)$ Å
$b = 7.2832\ (5)$ Å
$c = 16.3599\ (19)$ Å
$\beta = 101.298\ (9)^\circ$
$V = 949.24\ (17)$ Å$^3$
$Z = 2$

$D_x = 1.360$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 5493 reflections
$\theta = 2.5$–$27.9^\circ$
$\mu = 0.23$ mm$^{-1}$
$T = 293\ (2)$ K
Prism, colourless
$0.36 \times 0.31 \times 0.26$ mm

*Data collection*

Stoe IPDS-II diffractometer
$\omega$ scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{\min} = 0.936$, $T_{\max} = 0.964$
5547 measured reflections
3213 independent reflections

2127 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.055$
$\theta_{\max} = 26.0^\circ$
$h = -10 \rightarrow 10$
$k = -8 \rightarrow 7$
$l = -20 \rightarrow 19$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.105$
$S = 0.92$
3213 reflections
298 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.13$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.14$ e Å$^{-3}$
Extinction correction: *SHELXL97*
Extinction coefficient: 0.017 (3)
Absolute structure: Flack (1983), 1200 Friedel pairs
Flack parameter: 0.01 (8)

**Table 1**
Selected interatomic distances (Å).

| | | | |
|---|---|---|---|
| C1—N1 | 1.136 (5) | C15—O2 | 1.206 (4) |
| C2—N2 | 1.137 (5) | C15—O3 | 1.336 (4) |
| C5—O1 | 1.354 (4) | C16—O3 | 1.452 (4) |
| C9—O1 | 1.403 (3) | | |

**Table 2**
Hydrogen-bond geometry (Å, °).

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| C7—H7$\cdots$N1$^{\rm i}$ | 0.93 (4) | 2.45 (4) | 3.322 (5) | 155 (3) |

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

Friedel pairs were not merged. Atoms H20 and H22 were included in calculated positions and treated using a riding model [C—H(aromatic) = 0.93 Å, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(parent C atom)]. The other H atoms were found in a difference electron-density map and were refined with isotropic displacement parameters.

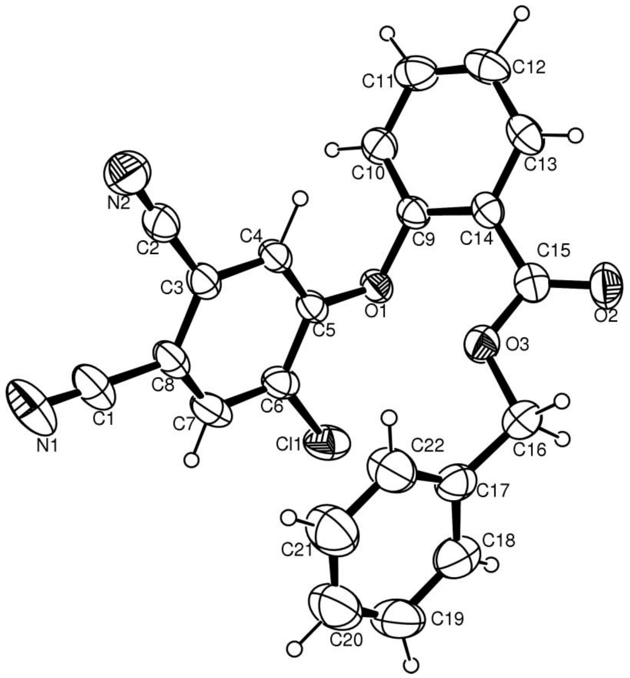


**Figure 1**
An *ORTEPIII* drawing of (I) showing the atomic numbering. Displacement ellipsoids of non-H atoms are drawn at the 30% probability level.

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

## References

Atalay, Ş., Ağar, A., Akdemir, N. & Ağar, E. (2003). *Acta Cryst.* E**59**, o1111–o1112.
Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Erdem, T. K., Atalay, Ş., Akdemir, N., Ağar, E. & Kantar, C. (2004). *Acta Cryst.* E**60**, o1849–o1850.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Iskeleli, N. O. & Ağar, A. (2005). *Acta Cryst.* E**61**, o158–o159.
Leznoff, C. C.& Lever, A. B. P. (1989–1996). *Phthalocyanines: Properties and Applications*, Vols 1, 2, 3 and 4. Weinheim/New York: VCH Publishers Inc.
McKeown, N. B. (1998). *Phthalocyanine Materials: Synthesis, Structure and Function*. Cambridge University Press.
Ocak, N., Büyükgüngör, O., Akdemir, N., Ağar, E., Özil, M. & Erdönmez, A. (2004). *Acta Cryst.* E**60**, o505–o507.
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.