Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

4-(3,4-Methylenedioxyphenoxy)phthalonitrile

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

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

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

Key indicators
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.127
Data-to-parameter ratio = 13.0

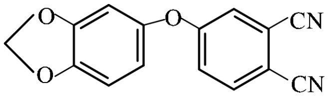
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 9 December 2004
Accepted 14 December 2004
Online 24 December 2004

In the structure of the title compound, $C_{15}H_8N_2O_3$, the phthalonitrile group and the 3,4-methylenedioxyphenoxy substituent are twisted with respect to one another by 58.89 (3)°. In the crystal structure, the molecules are stacked in the a-axis direction and are connected *via* weak C—H···N intermolecular contacts into chains, which extend in the direction of the c axis.

Comment

Substituted phthalonitriles are generally used for the synthesis of symmetrically and unsymmetrically peripherally and non-peripherally substituted phthalocyanines and subphthalocyanines (McKeown, 1998; Leznoff & Lever, 1989–1996). In addition to their extensive use as dyes and pigments, phthalocyanines have found widespread applications in catalysis, in optical recording, in photoconductive materials, in photodynamic therapy and as chemical sensors (Leznoff & Lever, 1989).



(I)

The molecule of the title compound, (I), is not planar (Fig. 1). The dihedral angle between the phthalonitrile moiety and the 3,4-methylenedioxyphenoxy group is 58.89 (3)°. The C—O and C—C bond distances in the 3,4-methylenedioxyphenoxy group are similar to values reported in the literature (Okamoto *et al.*, 1993. The lengths of the two C≡N triple bonds [C1≡N1 = 1.140 (2) Å and C2≡N2 = 1.143 (2) Å] are consistent with those found in similar compounds (Ocak *et al.*, 2003, 2004; Atalay *et al.*, 2003; Erdem *et al.*, 2004). In the crystal structure, the molecules are stacked in the a-axis direction and are connected *via* weak intermolecular C—H···N contacts into chains, which extend in the c-axis direction.

Experimental

3,4-Methylenedioxyphenol (0.96 g, 6.95 mmol) and 4-nitrophthalonitrile (1.00 g, 5.78 mmol) were dissolved in dry dimethylformamide (40 ml) and stirred under N_2. Dry fine-powdered sodium carbonate (1.06 g, 10.00 mmol) was added in portions (10 × 1 mmol) every 10 min. The reaction mixture was stirred for 48 h at room temperature and afterwards poured into ice-water (200 g). The product was filtered off and washed with NaOH solution (10 *w*/*w*) and water until the filtrate was neutral. Recrystallization from ethanol gave 0.84 g (55.04%) of the product. Single crystals were obtained at room temperature by slow evaporation of an ethanol solution (m.p. 408 K; elemental analysis calculated for $C_{15}H_8N_2O_3$: C 68.18, H 3.05, N 10.60%; found: C 68.16 H 3.08 N 10.64%).

Crystal data

$C_{15}H_8N_2O_3$
$M_r = 264.23$
Monoclinic, $P2_1/c$
$a = 4.0159$ (3) Å
$b = 23.6948$ (14) Å
$c = 12.7652$ (11) Å
$\beta = 91.642$ (7)°
$V = 1214.19$ (16) Å^3
$Z = 4$
$D_x = 1.445$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 22 043 reflections
$\theta = 1.6$–27.2°
$\mu = 0.10$ mm^{-1}
$T = 293$ (2) K
Prism, colourless
0.65 × 0.48 × 0.33 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: none
13 225 measured reflections
2363 independent reflections
1887 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.098$
$\theta_{\rm max} = 26.0°$
$h = -4 \rightarrow 4$
$k = -29 \rightarrow 29$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.127$
$S = 1.07$
2363 reflections
182 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0662P)^2 + 0.0684P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.13$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.099 (11)

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.140 (2)	C11—C12	1.375 (2)
C2—N2	1.143 (2)	C12—O3	1.3744 (19)
C6—O1	1.3637 (17)	C15—O3	1.413 (2)
C9—O1	1.4022 (17)	C15—O2	1.425 (2)
C11—O2	1.368 (2)		
C6—O1—C9	122.45 (11)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8···N2$^{\rm i}$	0.93	2.53	3.450 (2)	168

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

H atoms were placed in calculated positions and refined isotropically using a riding model [aromatic C—H = 0.93 Å and CH_2 C—H = 0.97 Å, and $U_{\rm iso}(H) = 1.2U_{\rm eq}(C)$].

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).

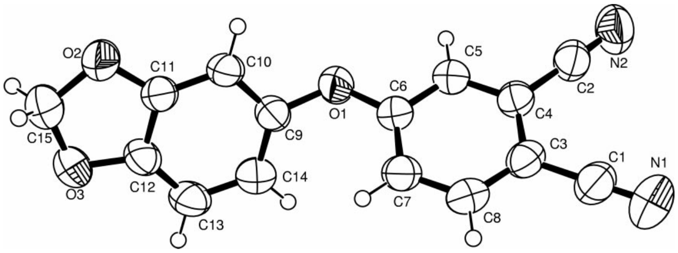


Figure 1
An *ORTEPIII* drawing (Burnett & Johnson, 1996) of the title compound, showing the atomic numbering and displacement ellipsoids drawn at the 50% probability level.

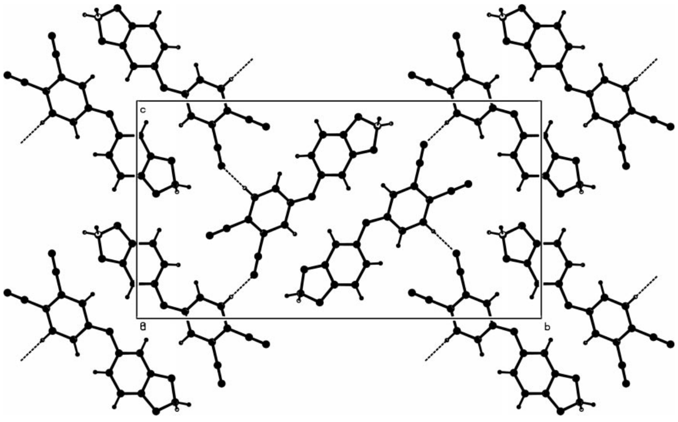


Figure 2
The crystal structure of the title compound, viewed along the *a* axis. The intermolecular C—H···N contacts are shown as dashed lines.

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.

Leznoff, C. C. & Lever, A. B. P. (1989). *Phthalocyanines: Properties and Applications*, Vol. 1. Weinheim, New York: VCH Publishers Inc.

McKeown, N. B. (1998). *Phthalocyanine Materials: Synthesis, Structure and Function*. Cambridge University Press.

Ocak, N., Ağar, A., Akdemir, N., Ağar, E., García-Granda, S. & Erdönmez, A. (2003). *Acta Cryst.* E**59**, o1000–o1001.

Ocak, N., Çoruh, U., Akdemir, N., Kantar, C., Ağar, E. & Erdönmez, A. (2004). *Acta Cryst.* E**60**, o33–o34.

Okamoto, K., Fujii, S. & Tomita, K. (1993). *Acta Cryst.* C**49**, 1125–1127.

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.