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4-(2-Bromo-4,5-dimethoxybenzyloxy)phthalonitrile

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Key indicators

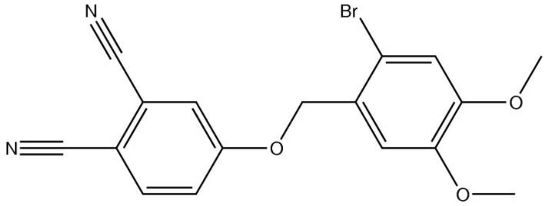
Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.127
Data-to-parameter ratio = 18.3

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

The title phthalonitrile derivative, $C_{17}H_{13}BrN_2O_3$, contains two aromatic rings, which are nearly coplanar. The crystal structure is stabilized by C—H···O hydrogen-bond interactions.

Comment

Monosubstituted phthalonitriles have been used as starting materials for symmetrically and unsymmetrically monosubstituted phthalocyanines and subphthalocyanines (McKeown, 1998), which are important components for dyes, pigments, gas sensors, optical limiters and liquid crystals, and which are also used in medicine, as singlet oxygen photosensitizers for photodynamic therapy (PDT) (Leznoff & Lever, 1989–1996).



(I)

The molecule of the title compound, (I), is shown in Fig. 1. The lengths of the two C≡N triple bonds (Table 1) are consistent with those found in similar compounds (Atalay *et al.*, 2003; Erdem *et al.*, 2004; Ískeleli & Ağar, 2005). The C—Br bond distance is close to the values reported for 4-(3-bromobenzylideneamino)-3-(4-chlorobenzyl)-4,5-dihydro-1*H*-1,2,4-triazol-5-one (Atalay *et al.*, 2004). The two aromatic rings in the molecule are nearly coplanar, the dihedral angle being 1.23 (3)°.

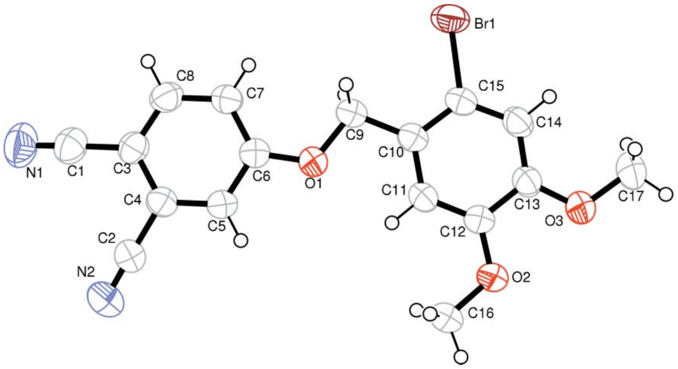


Figure 1
A drawing of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

The crystal structure of (I) is stabilized by inter- and intramolecular C—H⋯O hydrogen-bond interactions (Table 2).

Experimental

2-Bromo-4,5-dimethoxybenzyl alcohol (1.54 g, 6.23 mmol) and 4-nitrophthalonitrile (1.0 g, 5.78 mmol) were dissolved in dry dimethylformamide (40 ml) with stirring under N_2 at 323 K. Dry fine-powdered potassium carbonate (1.4 g, 10.00 mmol) was added in portions (10 × 1 mmol) every 10 min. The reaction mixture was stirred for 48 h at 323 K and then 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 a white product (yield 0.96 g, 44.431%). Single crystals of (I) were obtained from absolute ethanol at room temperature by slow evaporation (m.p. 429–431 K). Elemental analysis, calculated for $C_{17}H_{13}BrN_2O_3$: C 54.71, H 3.51, N 7.51%; found: C 54.66, H 3.50, N 7.58%.

Crystal data

$C_{17}H_{13}BrN_2O_3$
$M_r = 373.20$
Monoclinic, $P2_1/c$
$a = 8.7619\ (5)$ Å
$b = 9.8234\ (7)$ Å
$c = 18.5527\ (10)$ Å
$\beta = 92.270\ (4)^\circ$
$V = 1595.61\ (17)$ Å^3
$Z = 4$
$D_x = 1.554$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 23864 reflections
$\theta = 2.2$–28.0°
$\mu = 2.59$ mm^{-1}
$T = 296$ K
Prism, colourless
0.51 × 0.37 × 0.25 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{\min} = 0.782$, $T_{\max} = 0.898$
27104 measured reflections
3831 independent reflections
2815 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.084$
$\theta_{\max} = 28.0^\circ$
$h = -11 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.127$
$S = 1.11$
3831 reflections
209 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0723P)^2 + 0.0205P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.95$ e Å^{-3}
$\Delta\rho_{\min} = -0.74$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.033 (3)

Table 1
Selected bond lengths (Å).

C1—N1	1.136 (4)	C13—O3	1.362 (3)
C2—N2	1.141 (4)	C15—Br1	1.904 (3)
C6—O1	1.347 (3)	C16—O2	1.419 (3)
C9—O1	1.428 (3)	C17—O3	1.427 (4)
C12—O2	1.359 (3)		

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C8—H8⋯O3$^{\rm i}$	0.93	2.55	3.323 (4)	140
C11—H11⋯O1	0.93	2.28	2.655 (3)	103

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

H atoms were positioned geometrically and treated using a riding model, with aromatic C—H distances of 0.93 Å, methyl C—H distances of 0.96 Å and methylene C—H distances of 0.97 Å. $U_{\rm iso}$(H) values were set at $xU_{\rm eq}$(carrier atom), where $x = 1.5$ for methyl H and 1.2 for other H atoms.

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).

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