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4-(3-Methoxyphenylsulfanyl)phthalonitrile

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.057
wR factor = 0.159
Data-to-parameter ratio = 15.1

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

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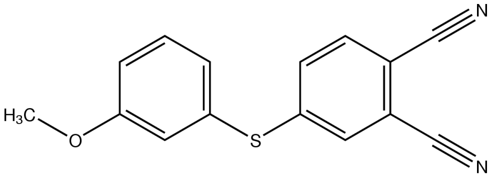
The title compound, $C_{15}H_{10}N_2OS$, is composed of a 3-methoxyphenylsulfanyl moiety connected *via* the S atom to a phthalonitrile group. The crystal structure is stabilized by C—H···N and C—H···O intermolecular hydrogen bonds.

Comment

Substituted phthalonitriles have been used as starting materials for phthalocyanines (McKeown, 1998). 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, 1989–1996).



(I)

The title compound, (I), contains two benzene rings (*A*: C3–C8; *B*: C9–C14), as shown in Fig. 1. The dihedral angles between rings *A* and *B* is 69.5 (1)°. S1 and O1 are displaced, on the same side, from the plane of ring *B* by 0.177 (1) and 0.018 (2) Å, respectively.

The bond lengths and angles for (I) are listed in Table 1. The triple-bond distances, 1.134 (4) and 1.144 (5) Å, for C1≡N1 and C2≡N2, respectively, agree with literature values (Atalay *et al.*, 2003, 2004; Du *et al.*, 2001). The geometry around the S atom also shows good agreement with literature values (Petek *et al.*, 2004).

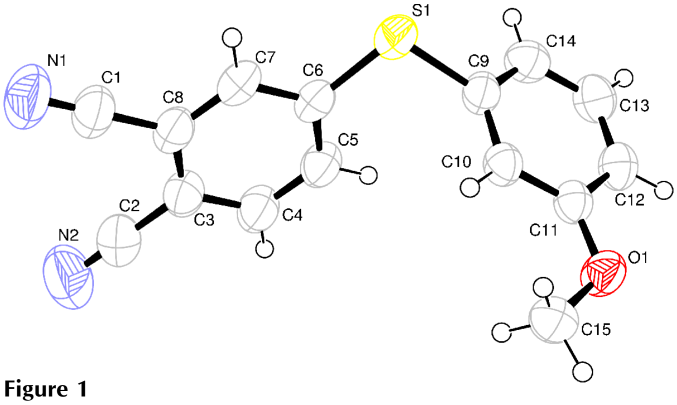


Figure 1
Perspective view of the molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

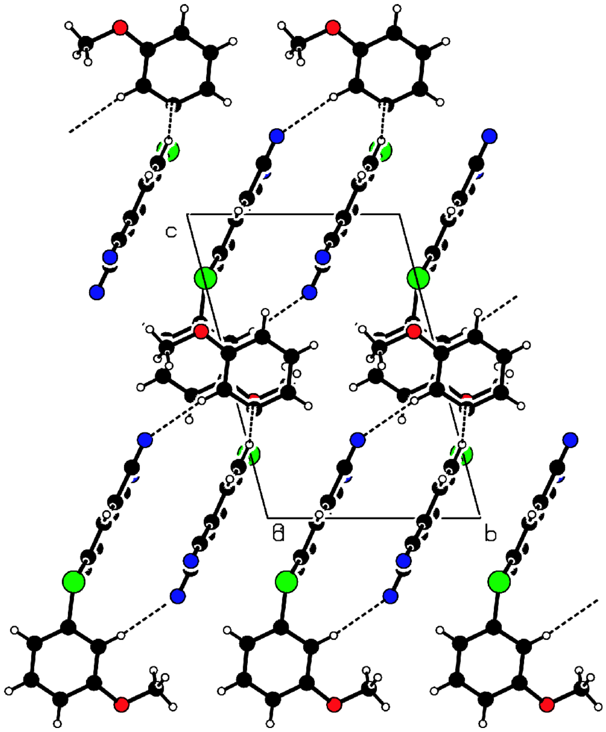


Figure 2
The molecular packing in (I), viewed along the *a* axis, showing the hydrogen-bonding interactions as dashed lines.

The crystal packing, shown in Fig. 2, is stabilized by two intermolecular hydrogen bonds: C10$\cdots$N1^{i} = 3.534 (4) Å and C5$\cdots$O1^{ii} = 3.191 (3) Å; details and symmetry codes are given in Table 2.

Experimental

3-Methoxybenzenethiol (0.89 g, 6.35 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 (1.0 g, 7.25 mmol) was added in portions (10 × 1 mmol) every 10 min. The reaction mixture was stirred for 48 h at room temperature and then poured into ice-water (100 g). The product was filtered off and washed with NaOH solution and water (10% *w*/*w*) until the filtrate was neutral. Recrystallization from ethanol gave a white product (yield 1.54 g, 81.17%). Single crystals were obtained from absolute ethanol at room temperature *via* slow evaporation (m.p. 370 K); elemental analysis calculated for $C_{15}H_{10}N_2OS$: C 67.65, H 3.78, N 10.52%; found: C 67.52, H 3.82, N 10.46%. ^{1}H NMR ($CDCl_3$): 3.84 (*s*, 3H, OCH_3), 7.04–7.62 p.p.m. (*m*, 7H, Ar). ^{13}C NMR ($CDCl_3$): 55.52 (OCH_3), 111.30, 115.06, 115.41, 116.28, 116.34, 120.27, 127.12, 129.30, 129.99, 130.17, 131.29, 133.28, 148.18, 160.82 p.p.m.

Crystal data

$C_{15}H_{10}N_2OS$
M_r = 266.31
Triclinic, $P\overline{1}$
a = 7.5982 (9) Å
b = 8.5208 (11) Å
c = 11.6213 (14) Å
α = 101.059 (10)°
β = 96.146 (9)°
γ = 113.501 (9)°
V = 662.94 (14) Å^3
Z = 2
D_x = 1.334 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 13254 reflections
θ = 2.7–29.0°
μ = 0.24 mm^{-1}
T = 293 (2) K
Plate, pale yellow
0.61 × 0.34 × 0.08 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: by integration (*X-SHAPE*; Stoe & Cie, 2002)
T_{min} = 0.898, T_{max} = 0.978
8721 measured reflections
2591 independent reflections
1714 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
θ_{max} = 26.0°
$h = -9 \rightarrow 9$
$k = -10 \rightarrow 10$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.159
S = 1.00
2591 reflections
172 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.099P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

C15—O1	1.426 (3)	S1—C6	1.757 (3)
O1—C11	1.358 (3)	S1—C9	1.781 (3)
N1—C1	1.134 (4)	C2—N2	1.144 (5)
C11—O1—C15	118.1 (2)	C6—S1—C9	104.59 (13)
C15—O1—C11—C10	7.2 (4)	C15—O1—C11—C12	−175.4 (3)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C10—H10$\cdots$N1^{i}	0.93	2.62	3.534 (4)	167
C5—H5$\cdots$O1^{ii}	0.93	2.44	3.191 (3)	138

Symmetry codes: (i) $-x, -1-y, -z$; (ii) $1-x, -y, 1-z$.

H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for C_{ar}—H, and C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for CH_3 groups.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PARST* (Nardelli, 1995).

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