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3-(2-Nitrophenoxy)phthalonitrile

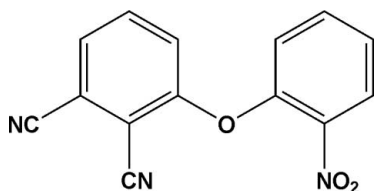
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.067; wR factor = 0.146; data-to-parameter ratio = 11.9.In the title compound, $\text{C}_{14}\text{H}_7\text{N}_3\text{O}_3$, the dihedral angle between the two arene units is 62.57 (12)°.

Related literature

For related literature, see: Atalay *et al.* (2003, 2004); Cave *et al.* (1986); Köysal *et al.* (2004); Leznoff & Lever (1989–1996); McKeown (1998); Ocak İskeleli (2007); Ocak *et al.* (2003), Sharman & van Lier (2003).

Experimental

Crystal data

 $\text{C}_{14}\text{H}_7\text{N}_3\text{O}_3$
 $M_r = 265.23$
 Monoclinic, $P2_1/n$
 $a = 8.0814$ (17) Å

 $b = 7.9899$ (12) Å
 $c = 19.068$ (3) Å
 $\beta = 95.944$ (15)°
 $V = 1224.6$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 295$ (2) K
 $0.4 \times 0.4 \times 0.1$ mm

Data collection

 Bruker *P4* diffractometer
 Absorption correction: none
 3018 measured reflections
 2155 independent reflections
 1252 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.092$
 3 standard reflections
 every 97 reflections
 intensity decay: none

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.067$
 $wR(F^2) = 0.145$
 $S = 1.03$
 2155 reflections

 181 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.20$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.27$ e Å⁻³

 Data collection: *XSCANS* (Bruker, 1997); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GD2031).

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supporting information

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3-(2-Nitrophenoxy)phthalonitrile

Xian-Fu Zhang, Dandan Jia, Aijun Song and Qiang Liu

S1. Comment

Phthalonitriles are among the most important precursors of phthalocyanine materials (Leznoff, 1989–1996).

Monophenoxyphthalonitriles have been used for preparing symmetrical phthalocyanines which have been applied in many areas, such as laser printing, photocopying, optical data storage, and catalysis (McKeown, 1998).

In the title compound, (I), (Fig. 1) the triple bond lengths between C and N, 1.136 (5) Å and 1.129 (5) Å, agree with literature values (Ocak *et al.*, 2003). The geometry around the O atoms is in good agreement with the literature (Atalay *et al.*, 2003, 2004; Köysal *et al.*, 2004). The dihedral angle between the two intramolecular arene moieties is 62.57 (12)°.

S2. Experimental

o-nitrophenol (1.39 g, 10.0 mmol) and 3-nitroththalonitrile (1.73 g, 10.0 mmol) were dissolved in dry DMF (15 ml) with stirring under N₂. Dry fine-powdered potassium carbonate (2.5 g, 18.1 mmol) was added over the course 1 h in equal portions every 10 min. The reaction mixture was stirred for 48 h at room temperature and poured into iced water (150 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 1.72 g, 65%). Single crystals were obtained from absolute ethanol at room temperature *via* slow evaporation (m.p. 397–400 K). IR data ($\nu_{\text{max}}/\text{cm}^{-1}$): 3050(Ar—H), 1591(NO₂), 2230(CN). NMR δ (H) 7.34–7.39(1H, m), 7.53–7.63(2H,m), 7.81–7.93(3H,m), 8.19–8.26(1H,m).

S3. Refinement

H atoms were included as riding atoms in geometrically idealized positions with C—H distances 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

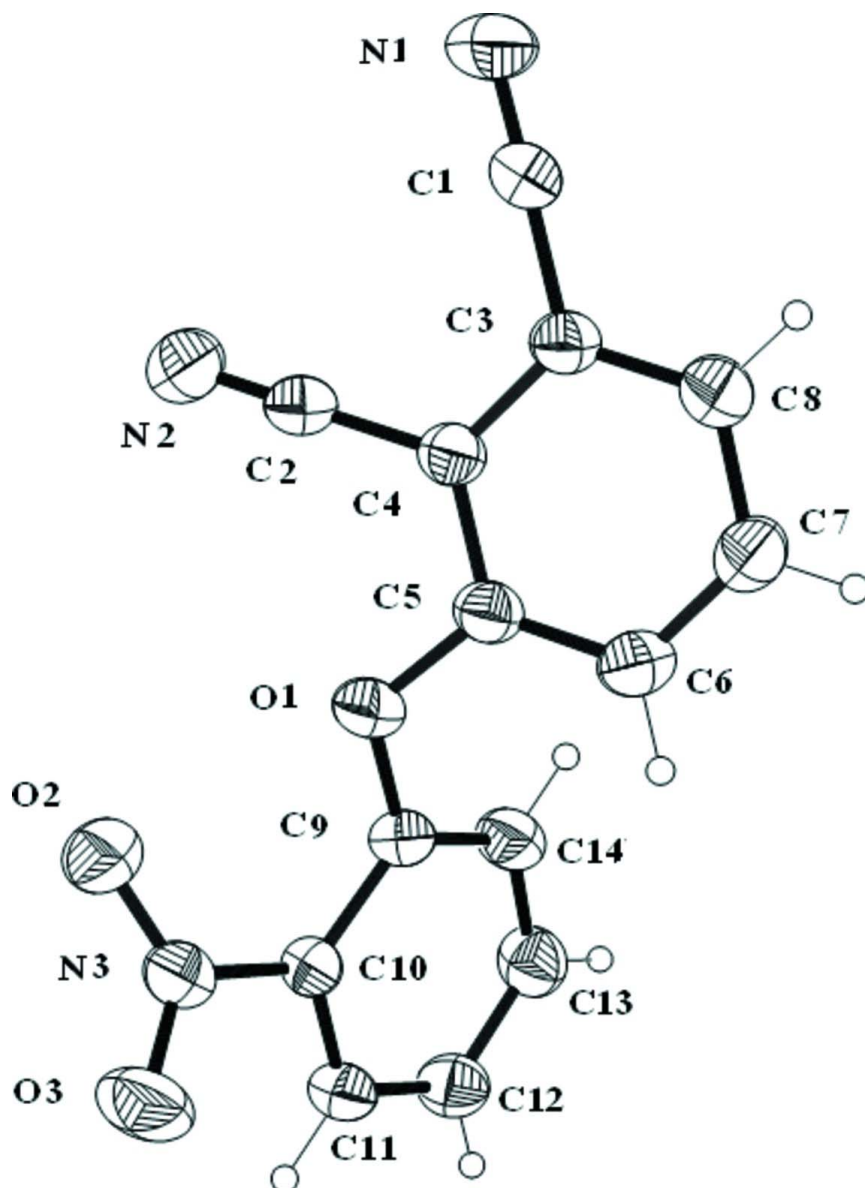


Figure 1

The molecular structure of $C_{14}H_7N_3O_3$ with 35% probability ellipsoids, showing the atom numbering scheme.

3-(2-Nitrophenoxy)phthalonitrile

Crystal data

$C_{14}H_7N_3O_3$

$M_r = 265.23$

Monoclinic, $P2_1/n$

Hall symbol: $-P 2_1n$

$a = 8.0814 (17) \text{ \AA}$

$b = 7.9899 (12) \text{ \AA}$

$c = 19.068 (3) \text{ \AA}$

$\beta = 95.944 (15)^\circ$

$V = 1224.6 (4) \text{ \AA}^3$

$Z = 4$

$F(000) = 544$

$D_x = 1.439 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 51 reflections

$\theta = 5.0\text{--}12.5^\circ$

$\mu = 0.11 \text{ mm}^{-1}$

$T = 295 \text{ K}$

Plate, colorless

$0.4 \times 0.4 \times 0.1 \text{ mm}$

Data collection

Bruker P4 diffractometer	$R_{\text{int}} = 0.092$
Radiation source: fine-focus sealed tube	$\theta_{\text{max}} = 25.0^\circ, \theta_{\text{min}} = 2.2^\circ$
Graphite monochromator	$h = -1 \rightarrow 9$
ω scans	$k = -9 \rightarrow 1$
3018 measured reflections	$l = -22 \rightarrow 22$
2155 independent reflections	3 standard reflections every 97 reflections
1252 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.067$	H-atom parameters constrained
$wR(F^2) = 0.146$	$w = 1/[\sigma^2(F_o^2) + (0.001P)^2 + 1.2P]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
2155 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
181 parameters	$\Delta\rho_{\text{max}} = 0.20 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.27 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.4115 (3)	0.5913 (3)	0.63769 (12)	0.0683 (8)
O2	0.5975 (4)	0.8586 (4)	0.66974 (15)	0.0806 (9)
O3	0.7869 (4)	0.8009 (4)	0.75304 (16)	0.0932 (11)
N1	0.0726 (6)	0.4464 (5)	0.36401 (19)	0.0967 (14)
N2	0.1821 (5)	0.7972 (5)	0.50408 (18)	0.0822 (11)
N3	0.6493 (4)	0.7811 (4)	0.72224 (17)	0.0609 (9)
C1	0.1511 (5)	0.4108 (5)	0.4142 (2)	0.0651 (11)
C2	0.2279 (5)	0.6648 (6)	0.51586 (18)	0.0566 (10)
C3	0.2487 (5)	0.3691 (5)	0.47972 (18)	0.0534 (9)
C4	0.2840 (4)	0.4955 (5)	0.53013 (17)	0.0492 (9)
C5	0.3749 (4)	0.4568 (5)	0.59366 (18)	0.0524 (9)
C6	0.4322 (5)	0.2954 (5)	0.6072 (2)	0.0623 (10)
H6A	0.4952	0.2702	0.6495	0.075*
C7	0.3950 (5)	0.1729 (5)	0.5576 (2)	0.0657 (11)
H7A	0.4328	0.0644	0.5667	0.079*
C8	0.3024 (5)	0.2082 (5)	0.49420 (19)	0.0620 (10)

H8A	0.2765	0.1234	0.4615	0.074*
C9	0.4213 (4)	0.5689 (5)	0.71071 (17)	0.0523 (9)
C10	0.5397 (4)	0.6628 (4)	0.75215 (18)	0.0480 (9)
C11	0.5535 (5)	0.6431 (5)	0.82497 (18)	0.0584 (10)
H11A	0.6334	0.7036	0.8530	0.070*
C12	0.4516 (5)	0.5362 (5)	0.85555 (19)	0.0603 (10)
H12A	0.4628	0.5220	0.9042	0.072*
C14	0.3162 (5)	0.4646 (5)	0.7422 (2)	0.0626 (11)
H14A	0.2348	0.4043	0.7148	0.075*
C13	0.3319 (5)	0.4496 (5)	0.8141 (2)	0.0643 (11)
H13A	0.2599	0.3792	0.8352	0.077*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.090 (2)	0.0637 (18)	0.0462 (14)	-0.0149 (15)	-0.0149 (13)	-0.0003 (13)
O2	0.090 (2)	0.082 (2)	0.0670 (18)	-0.0169 (17)	-0.0075 (16)	0.0199 (16)
O3	0.0672 (19)	0.119 (3)	0.088 (2)	-0.0375 (19)	-0.0167 (17)	0.0071 (19)
N1	0.128 (4)	0.087 (3)	0.065 (2)	0.014 (3)	-0.036 (2)	-0.007 (2)
N2	0.113 (3)	0.067 (3)	0.063 (2)	0.008 (2)	-0.005 (2)	0.0033 (19)
N3	0.064 (2)	0.062 (2)	0.0549 (19)	-0.0103 (18)	-0.0035 (17)	-0.0013 (17)
C1	0.082 (3)	0.056 (2)	0.054 (2)	-0.005 (2)	-0.009 (2)	-0.0072 (19)
C2	0.065 (3)	0.062 (3)	0.0408 (19)	-0.002 (2)	-0.0030 (18)	-0.0008 (19)
C3	0.060 (2)	0.056 (2)	0.0429 (19)	-0.005 (2)	-0.0001 (17)	0.0012 (18)
C4	0.049 (2)	0.053 (2)	0.0451 (19)	-0.0043 (18)	0.0015 (15)	-0.0005 (17)
C5	0.056 (2)	0.056 (2)	0.0438 (19)	-0.006 (2)	0.0004 (17)	-0.0018 (18)
C6	0.064 (2)	0.068 (3)	0.053 (2)	0.001 (2)	-0.0055 (19)	0.006 (2)
C7	0.080 (3)	0.057 (2)	0.059 (2)	0.007 (2)	0.002 (2)	0.006 (2)
C8	0.076 (3)	0.057 (3)	0.053 (2)	0.000 (2)	0.004 (2)	-0.0059 (19)
C9	0.056 (2)	0.053 (2)	0.045 (2)	0.0009 (19)	-0.0076 (17)	0.0009 (17)
C10	0.049 (2)	0.043 (2)	0.050 (2)	0.0003 (17)	-0.0030 (16)	-0.0021 (16)
C11	0.062 (2)	0.059 (2)	0.051 (2)	-0.002 (2)	-0.0103 (19)	-0.0059 (18)
C12	0.065 (3)	0.068 (3)	0.047 (2)	-0.001 (2)	0.0039 (19)	-0.0027 (19)
C14	0.059 (2)	0.065 (3)	0.061 (2)	-0.016 (2)	-0.007 (2)	0.001 (2)
C13	0.066 (3)	0.065 (3)	0.062 (2)	-0.011 (2)	0.010 (2)	0.002 (2)

Geometric parameters (Å, °)

O1—C5	1.377 (4)	C6—H6A	0.9300
O1—C9	1.398 (4)	C7—C8	1.383 (5)
O2—N3	1.214 (4)	C7—H7A	0.9300
O3—N3	1.213 (4)	C8—H8A	0.9300
N1—C1	1.129 (5)	C9—C14	1.372 (5)
N2—C2	1.136 (5)	C9—C10	1.395 (5)
N3—C10	1.452 (5)	C10—C11	1.390 (5)
C1—C3	1.445 (5)	C11—C12	1.359 (5)
C2—C4	1.443 (6)	C11—H11A	0.9300
C3—C8	1.376 (5)	C12—C13	1.372 (5)

C3—C4	1.403 (5)	C12—H12A	0.9300
C4—C5	1.385 (5)	C14—C13	1.369 (5)
C5—C6	1.385 (5)	C14—H14A	0.9300
C6—C7	1.372 (5)	C13—H13A	0.9300
C5—O1—C9	119.6 (3)	C3—C8—C7	119.8 (3)
O3—N3—O2	123.6 (4)	C3—C8—H8A	120.1
O3—N3—C10	117.4 (3)	C7—C8—H8A	120.1
O2—N3—C10	119.0 (3)	C14—C9—C10	119.9 (3)
N1—C1—C3	178.2 (5)	C14—C9—O1	122.7 (3)
N2—C2—C4	179.1 (4)	C10—C9—O1	117.4 (3)
C8—C3—C4	119.8 (3)	C11—C10—C9	119.0 (4)
C8—C3—C1	121.4 (3)	C11—C10—N3	118.5 (3)
C4—C3—C1	118.7 (3)	C9—C10—N3	122.5 (3)
C5—C4—C3	119.3 (3)	C12—C11—C10	120.6 (3)
C5—C4—C2	120.1 (3)	C12—C11—H11A	119.7
C3—C4—C2	120.5 (3)	C10—C11—H11A	119.7
O1—C5—C4	114.8 (3)	C11—C12—C13	119.5 (4)
O1—C5—C6	124.5 (3)	C11—C12—H12A	120.3
C4—C5—C6	120.5 (3)	C13—C12—H12A	120.3
C7—C6—C5	119.4 (3)	C13—C14—C9	119.6 (4)
C7—C6—H6A	120.3	C13—C14—H14A	120.2
C5—C6—H6A	120.3	C9—C14—H14A	120.2
C6—C7—C8	121.1 (4)	C14—C13—C12	121.3 (4)
C6—C7—H7A	119.4	C14—C13—H13A	119.3
C8—C7—H7A	119.4	C12—C13—H13A	119.3
