Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

3,3′-(*m*-Phenylenedioxy)diphthalonitrile

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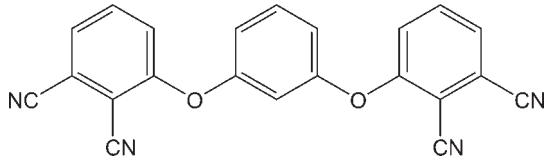
Received 13 March 2010; accepted 26 March 2010

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.039; wR factor = 0.111; data-to-parameter ratio = 15.7.

In the title compound, $C_{22}H_{10}N_4O_2$, the dihedral angles between the mean planes of the central benzene ring and the pendant rings are 79.20 (6) and 80.29 (6)°. The dihedral angle between the pendant rings is 10.27 (7)°.

Related literature

For background to 'semi-rigid' molecules as ligands, see: Wang *et al.* (2005, 2009). For related structures, see: Huang *et al.* (2005); Zhang & Lu (2007).



Experimental

Crystal data

$C_{22}H_{10}N_4O_2$
M_r = 362.34
Monoclinic, $C2/c$
a = 15.668 (3) Å
b = 12.722 (3) Å
c = 19.004 (5) Å
β = 109.911 (6)°
V = 3561.7 (14) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 K
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Siemens, 1996)
T_{min} = 0.982, T_{max} = 0.991
10307 measured reflections
3992 independent reflections
3145 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.111
S = 1.03
3992 reflections
254 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

This work was supported by Professor Yongzhong Bian, the Doctoral Foundation of Shandong (grant No. 2007BS04027) and Postdoctoral Scientific Foundation of China and Shandong (grant Nos. 200603070 and 20070411093).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5363).

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supporting information

Acta Cryst. (2010). E**66**, o1011 [https://doi.org/10.1107/S1600536810011578]

3,3′-(*m*-Phenylenedioxy)diphthalonitrile

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S1. Comment

In the past few years, the semirigidity of molecules have been extensively employed for search of novel functional compounds. For example,a new family of multidentate O-donor ligands with a semirigid V-shaped molecular framework have been used to construct metal-organic coordination frameworks (Wang *et al.*, 2009; Wang *et al.*, 2005), in which some showed interesting properties. Here, we present the structure of a new semirigid organic ligand.

The crystal structure of the title compound is given in Fig. 1. As can be found, all the bond lengths and angles are normal and correspond to those observed in related compound (Huang *et al.*, 2005; Zhang *et al.*, 2007). The aromatic rings (C3—C8 and C15—C20) in sides of the molecule are in the same direction of the aromatic rings(C9—C14) with a cis configuration. The three dihedral angles in the title compound are 79.81Å for C3—C8 and C9—C14, 80.83Å for C15—C20 and C9—C14,and 10.54 Å for C3—C8 and C15—C20, respectively.

S2. Experimental

Resorcinol (0.53 g, 5 mmol) and anhydrous K_2CO_3 was added to the solution of 2,3-dicyanophenyl nitrate (1.73 g, 10 mmol) in DMSO (25 ml). A kind of brown solution was generated after the solution was stirred for 48 hours at room temperature. The brown solution was added to 200 ml water, and was stirred for 30 min at room temperature. The precipitate formed was filtered, and washed by water. Yellow rods of (I) were obtained by solvent evaporation of the solution of the title compound in acetonitrile. Yield: 1.65 g, 91.2% Anal. for: $C_{22}H_{10}N_4O_2$ Calc. C, 72.92; H, 2.76; N, 15.47; Found: C, 72.85; H, 2.88; N, 15.44.

S3. Refinement

All H atoms were placed in geometrically idealized positions and treated as riding on their parent atoms with C(sp_2 hybrid)-H distances of 0.93Å (U_{iso}(H)=1.2U_{eq}(C)).

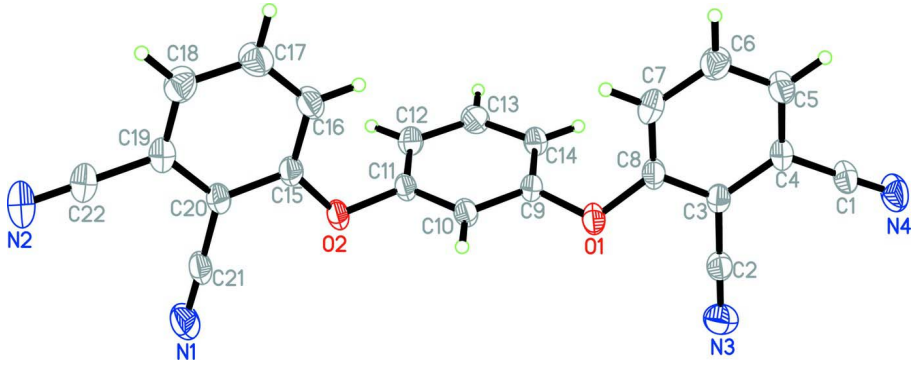

Figure 1

The molecular structure of (I). Displacement ellipsoids are drawn at 30% probability level.

3,3′-(*m*-Phenylenedioxy)diphthalonitrile

Crystal data

$C_{22}H_{10}N_4O_2$
$M_r = 362.34$
Monoclinic, $C2/c$
Hall symbol: -C 2yc
$a = 15.668$ (3) Å
$b = 12.722$ (3) Å
$c = 19.004$ (5) Å
$\beta = 109.911$ (6)°
$V = 3561.7$ (14) Å^3
$Z = 8$
$F(000) = 1488$
$D_x = 1.351$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4716 reflections
$\theta = 2.6$–27.4°
$\mu = 0.09$ mm^{-1}
$T = 298$ K
Rod, yellow
0.20 × 0.15 × 0.10 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Siemens, 1996)
$T_{min} = 0.982$, $T_{max} = 0.991$
10307 measured reflections
3992 independent reflections
3145 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 27.6°$, $\theta_{min} = 2.1°$
$h = -14\rightarrow20$
$k = -15\rightarrow15$
$l = -24\rightarrow23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.111$
$S = 1.03$
3992 reflections
254 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0506P)^2 + 1.0114P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0032 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C2	0.24878 (8)	0.23670 (11)	0.58469 (7)	0.0496 (3)
O1	0.41988 (6)	0.16069 (8)	0.64684 (5)	0.0597 (3)
O2	0.73624 (5)	0.20407 (7)	0.70914 (6)	0.0582 (3)
C9	0.49526 (7)	0.15184 (9)	0.62398 (7)	0.0425 (3)
C10	0.57791 (7)	0.17608 (9)	0.67719 (7)	0.0437 (3)
H10	0.5826	0.1926	0.7260	0.052*
C20	0.88271 (8)	0.16165 (10)	0.78616 (7)	0.0438 (3)
C11	0.65301 (7)	0.17492 (9)	0.65542 (7)	0.0459 (3)
C14	0.48704 (8)	0.12738 (10)	0.55183 (7)	0.0474 (3)
H14	0.4308	0.1110	0.5168	0.057*
C3	0.26451 (7)	0.12567 (10)	0.58820 (6)	0.0420 (3)
C4	0.19286 (7)	0.05388 (10)	0.56020 (6)	0.0443 (3)
C15	0.79734 (7)	0.12689 (9)	0.74065 (7)	0.0448 (3)
C12	0.64815 (9)	0.15132 (11)	0.58405 (8)	0.0554 (3)
H12	0.7000	0.1512	0.5707	0.067*
N3	0.23572 (10)	0.32515 (11)	0.58276 (8)	0.0727 (4)
C21	0.90124 (8)	0.27171 (11)	0.79679 (8)	0.0535 (3)
C8	0.35196 (8)	0.08751 (11)	0.61986 (7)	0.0466 (3)
C16	0.77924 (9)	0.02090 (10)	0.73059 (8)	0.0558 (3)
H16	0.7222	−0.0022	0.7005	0.067*
C5	0.20892 (9)	−0.05263 (11)	0.56674 (8)	0.0528 (3)
H5	0.1612	−0.1000	0.5485	0.063*
C19	0.95031 (8)	0.08725 (11)	0.82021 (7)	0.0499 (3)
C13	0.56427 (9)	0.12762 (11)	0.53235 (8)	0.0543 (3)
H13	0.5597	0.1115	0.4835	0.065*
N2	1.11044 (8)	0.15377 (13)	0.89675 (8)	0.0774 (4)
C1	0.10262 (8)	0.09249 (11)	0.52163 (8)	0.0524 (3)
C6	0.29672 (9)	−0.08846 (11)	0.60074 (8)	0.0585 (3)
H6	0.3077	−0.1604	0.6061	0.070*
C18	0.93170 (10)	−0.01859 (12)	0.80996 (8)	0.0630 (4)
H18	0.9762	−0.0681	0.8328	0.076*
C17	0.84594 (11)	−0.05017 (12)	0.76525 (9)	0.0661 (4)
H17	0.8332	−0.1216	0.7585	0.079*
C22	1.03960 (9)	0.12385 (12)	0.86413 (8)	0.0577 (4)
N4	0.03136 (8)	0.12171 (11)	0.48961 (8)	0.0725 (4)
C7	0.36803 (9)	−0.01905 (12)	0.62679 (8)	0.0564 (3)
H7	0.4269	−0.0441	0.6490	0.068*
N1	0.91780 (9)	0.35902 (11)	0.80612 (9)	0.0809 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C2	0.0399 (6)	0.0575 (8)	0.0501 (7)	−0.0042 (6)	0.0137 (5)	−0.0057 (6)
O1	0.0322 (4)	0.0797 (7)	0.0681 (6)	−0.0119 (4)	0.0181 (4)	−0.0269 (5)
O2	0.0293 (4)	0.0448 (5)	0.0869 (7)	−0.0052 (3)	0.0020 (4)	−0.0088 (4)

C9	0.0287 (5)	0.0456 (6)	0.0512 (7)	−0.0002 (4)	0.0111 (5)	−0.0022 (5)
C10	0.0347 (6)	0.0455 (6)	0.0463 (6)	−0.0043 (5)	0.0080 (5)	−0.0018 (5)
C20	0.0333 (6)	0.0515 (7)	0.0468 (6)	−0.0069 (5)	0.0137 (5)	−0.0004 (5)
C11	0.0286 (5)	0.0389 (6)	0.0635 (8)	−0.0048 (4)	0.0071 (5)	−0.0023 (5)
C14	0.0357 (6)	0.0514 (7)	0.0482 (7)	−0.0012 (5)	0.0056 (5)	−0.0031 (5)
C3	0.0334 (5)	0.0526 (7)	0.0406 (6)	−0.0027 (5)	0.0134 (5)	−0.0007 (5)
C4	0.0323 (6)	0.0554 (7)	0.0442 (6)	−0.0035 (5)	0.0117 (5)	0.0026 (5)
C15	0.0326 (5)	0.0463 (7)	0.0540 (7)	−0.0043 (5)	0.0129 (5)	−0.0004 (5)
C12	0.0422 (7)	0.0545 (7)	0.0764 (9)	−0.0084 (6)	0.0290 (6)	−0.0085 (7)
N3	0.0745 (9)	0.0599 (8)	0.0802 (9)	0.0003 (7)	0.0219 (7)	−0.0093 (6)
C21	0.0306 (6)	0.0589 (8)	0.0642 (8)	−0.0086 (5)	0.0076 (5)	−0.0030 (6)
C8	0.0313 (5)	0.0621 (8)	0.0462 (6)	−0.0049 (5)	0.0128 (5)	−0.0072 (5)
C16	0.0443 (7)	0.0484 (7)	0.0661 (8)	−0.0098 (6)	0.0077 (6)	0.0010 (6)
C5	0.0432 (7)	0.0544 (8)	0.0602 (8)	−0.0089 (6)	0.0168 (6)	0.0020 (6)
C19	0.0383 (6)	0.0640 (8)	0.0453 (6)	−0.0011 (6)	0.0116 (5)	0.0054 (6)
C13	0.0537 (7)	0.0589 (8)	0.0534 (7)	−0.0065 (6)	0.0223 (6)	−0.0070 (6)
N2	0.0416 (7)	0.1071 (11)	0.0731 (8)	−0.0068 (7)	0.0059 (6)	0.0123 (8)
C1	0.0369 (6)	0.0569 (8)	0.0581 (7)	−0.0078 (6)	0.0094 (6)	0.0031 (6)
C6	0.0523 (8)	0.0527 (8)	0.0718 (9)	0.0046 (6)	0.0229 (7)	0.0087 (7)
C18	0.0578 (8)	0.0600 (9)	0.0630 (8)	0.0099 (7)	0.0098 (7)	0.0136 (7)
C17	0.0674 (9)	0.0459 (7)	0.0737 (9)	−0.0043 (6)	0.0093 (8)	0.0075 (7)
C22	0.0395 (7)	0.0768 (10)	0.0532 (7)	0.0029 (6)	0.0111 (6)	0.0111 (7)
N4	0.0399 (6)	0.0705 (8)	0.0914 (10)	−0.0021 (6)	0.0018 (6)	0.0083 (7)
C7	0.0360 (6)	0.0694 (9)	0.0617 (8)	0.0092 (6)	0.0140 (6)	0.0056 (7)
N1	0.0538 (7)	0.0594 (8)	0.1145 (12)	−0.0151 (6)	0.0094 (8)	−0.0088 (8)

Geometric parameters (Å, º)

C2—N3	1.1421 (19)	C15—C16	1.3777 (18)
C2—C3	1.4315 (19)	C12—C13	1.3798 (19)
O1—C8	1.3750 (15)	C12—H12	0.9300
O1—C9	1.3949 (14)	C21—N1	1.1406 (19)
O2—C15	1.3596 (15)	C8—C7	1.377 (2)
O2—C11	1.4048 (14)	C16—C17	1.369 (2)
C9—C14	1.3686 (17)	C16—H16	0.9300
C9—C10	1.3795 (16)	C5—C6	1.3828 (19)
C10—C11	1.3740 (16)	C5—H5	0.9300
C10—H10	0.9300	C19—C18	1.377 (2)
C20—C15	1.3946 (16)	C19—C22	1.4406 (18)
C20—C19	1.4035 (18)	C13—H13	0.9300
C20—C21	1.4303 (19)	N2—C22	1.1369 (18)
C11—C12	1.3657 (19)	C1—N4	1.1368 (16)
C14—C13	1.3808 (18)	C6—C7	1.377 (2)
C14—H14	0.9300	C6—H6	0.9300
C3—C8	1.3834 (16)	C18—C17	1.382 (2)
C3—C4	1.4038 (16)	C18—H18	0.9300
C4—C5	1.3761 (19)	C17—H17	0.9300
C4—C1	1.4393 (17)	C7—H7	0.9300

N3—C2—C3	178.96 (15)	N1—C21—C20	178.61 (16)
C8—O1—C9	117.33 (9)	O1—C8—C7	122.54 (11)
C15—O2—C11	118.00 (9)	O1—C8—C3	116.79 (12)
C14—C9—C10	121.99 (11)	C7—C8—C3	120.59 (11)
C14—C9—O1	121.98 (10)	C17—C16—C15	119.49 (12)
C10—C9—O1	115.89 (11)	C17—C16—H16	120.3
C11—C10—C9	117.60 (11)	C15—C16—H16	120.3
C11—C10—H10	121.2	C4—C5—C6	119.28 (12)
C9—C10—H10	121.2	C4—C5—H5	120.4
C15—C20—C19	119.07 (12)	C6—C5—H5	120.4
C15—C20—C21	120.25 (11)	C18—C19—C20	120.26 (12)
C19—C20—C21	120.67 (11)	C18—C19—C22	121.00 (13)
C12—C11—C10	122.52 (11)	C20—C19—C22	118.73 (13)
C12—C11—O2	120.25 (11)	C12—C13—C14	121.25 (12)
C10—C11—O2	117.16 (11)	C12—C13—H13	119.4
C9—C14—C13	118.43 (11)	C14—C13—H13	119.4
C9—C14—H14	120.8	N4—C1—C4	178.29 (16)
C13—C14—H14	120.8	C7—C6—C5	120.85 (13)
C8—C3—C4	118.83 (12)	C7—C6—H6	119.6
C8—C3—C2	119.73 (11)	C5—C6—H6	119.6
C4—C3—C2	121.43 (10)	C19—C18—C17	119.06 (13)
C5—C4—C3	120.56 (11)	C19—C18—H18	120.5
C5—C4—C1	119.98 (11)	C17—C18—H18	120.5
C3—C4—C1	119.41 (12)	C16—C17—C18	121.77 (14)
O2—C15—C16	124.39 (11)	C16—C17—H17	119.1
O2—C15—C20	115.27 (10)	C18—C17—H17	119.1
C16—C15—C20	120.34 (11)	N2—C22—C19	177.82 (15)
C11—C12—C13	118.21 (12)	C8—C7—C6	119.83 (12)
C11—C12—H12	120.9	C8—C7—H7	120.1
C13—C12—H12	120.9	C6—C7—H7	120.1

C8—O1—C9—C14	41.14 (17)	C9—O1—C8—C3	−129.48 (12)
C8—O1—C9—C10	−143.02 (12)	C4—C3—C8—O1	−179.70 (10)
C14—C9—C10—C11	−0.04 (18)	C2—C3—C8—O1	0.21 (16)
O1—C9—C10—C11	−175.87 (11)	C4—C3—C8—C7	−2.83 (18)
C9—C10—C11—C12	0.24 (18)	C2—C3—C8—C7	177.08 (12)
C9—C10—C11—O2	177.36 (10)	O2—C15—C16—C17	−179.83 (13)
C15—O2—C11—C12	−77.49 (16)	C20—C15—C16—C17	−0.4 (2)
C15—O2—C11—C10	105.33 (13)	C3—C4—C5—C6	−0.51 (19)
C10—C9—C14—C13	−0.24 (19)	C1—C4—C5—C6	176.87 (12)
O1—C9—C14—C13	175.35 (12)	C15—C20—C19—C18	−1.39 (19)
C8—C3—C4—C5	2.48 (17)	C21—C20—C19—C18	179.66 (13)
C2—C3—C4—C5	−177.43 (11)	C15—C20—C19—C22	176.97 (12)
C8—C3—C4—C1	−174.92 (11)	C21—C20—C19—C22	−1.98 (18)
C2—C3—C4—C1	5.17 (17)	C11—C12—C13—C14	−0.1 (2)
C11—O2—C15—C16	−8.46 (19)	C9—C14—C13—C12	0.3 (2)
C11—O2—C15—C20	172.12 (11)	C4—C5—C6—C7	−1.1 (2)

C19—C20—C15—O2	−179.22 (11)	C20—C19—C18—C17	0.6 (2)
C21—C20—C15—O2	−0.27 (17)	C22—C19—C18—C17	−177.77 (14)
C19—C20—C15—C16	1.32 (19)	C15—C16—C17—C18	−0.4 (2)
C21—C20—C15—C16	−179.72 (13)	C19—C18—C17—C16	0.4 (2)
C10—C11—C12—C13	−0.2 (2)	O1—C8—C7—C6	177.91 (12)
O2—C11—C12—C13	−177.18 (11)	C3—C8—C7—C6	1.2 (2)
C9—O1—C8—C7	53.72 (17)	C5—C6—C7—C8	0.8 (2)