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3,3′-Difluoro-4,4′-(*p*-phenylenedioxy)-dibenzonitrile

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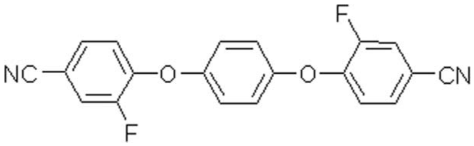
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.047; wR factor = 0.137; data-to-parameter ratio = 12.8.

The title compound, $C_{20}H_{10}F_2N_2O_2$, was synthesized from hydroquinone and 3,4-difluorobenzonitrile. The centroid of the central aromatic ring is on a crystallographic center of inversion. The dihedral angle between the central and terminal rings is 77.8 (3)°. In the crystal, chains linked by C—H···N bond occur.

Related literature

For the herbicidal actvity of hydroquinone derivatives, see: Bao *et al.* (2007). For related structures, see: Sørensen & Stuhr-Hansen (2009); Luo *et al.* (2009); Liu (2002).



Experimental

Crystal data

$C_{20}H_{10}F_2N_2O_2$
M_r = 348.30
Triclinic, $P\bar{1}$
a = 6.980 (1) Å
b = 7.615 (1) Å
c = 8.294 (1) Å
α = 106.376 (3)°
β = 93.698 (3)°
γ = 109.085 (3)°
V = 393.7 (1) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.42 × 0.37 × 0.32 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996) T_{min} = 0.782, T_{max} = 1.000
2165 measured reflections
1529 independent reflections
1259 reflections with $I > 2\sigma(I)$
R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.137
S = 1.07
1529 reflections
119 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C9—H9···N1^i	0.93	2.50	3.410 (2)	166

Symmetry code: (i) $-x, -y, -z+2$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SHELXTL* (Sheldrick, 2008); 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* (Sheldrick, 2008).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2138).

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supplementary materials

Acta Cryst. (2009). E**65**, o2340 [**doi:10.1107/S1600536809035247**]

3,3'-Difluoro-4,4'-(*p*-phenylenedioxy)dibenzonitrile

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Comment

There has been growing interest in the study of hydroquinone derivatives which are important intermediates in the synthesis of herbicides (Liu, 2002. Bao *et al.*, 2007). Only a few compounds of this kind have been structurally characterized so far. As part of our studies, we have synthesized the title compound from hydroquinone and 3,4-difluorobenzonitrile and report it's crystal structure in this article.

The crystal structure of the title compound (Fig. 1) utilizes the symmetry of the crystallographic inversion center similarly to a related selenium compound (Sørensen *et al.*, 2009). The two terminal (C1—C7) phenyl ring and the central ring together with the attached oxygen (C8—C10/O1) form three planes. Due to crystallograhic symmetry the two terminal phenyl rings are coplanar. The terminal (C1—C7) phenyl ring plane and the central ring plane enclose a dihedral angle of 77.8 (3)°. Otherwise, the molecule is bent with the C2—O1—C8 angle of 118.25°.

In the crystal structure, intermolecular C—H···N hydrogen bonds (Tab.1) connect neighboring molecules with each other to form a one-dimensional chain that stretches along the *c* axis (Fig.2).

Experimental

A DMF (10 ml) solution of hydroquinone (1 mmol) and 3,4-difluorobenzonitrile (2 mmol) was heated to 70°C in the presence of KOH and stirred for 37 h. Then the mixture was washed with water (30 ml) and extracted with ethyl acetate (three times). The organic solvent was removed under reduced pressure. Afterwards the product was purified by column chromatography on silica (pentane - ethyl acetate mixtures). Single crystals were obtained by slow evaporation of the solvent of an ethanolic solution at room temperature.

Refinement

H atoms were placed in calculated positions with C—H=0.93 Å. All H atoms were included in the final cycles of refinement using a riding model, with U_{iso}(H)=1.2U_{eq} of the carrier atoms.

Figures

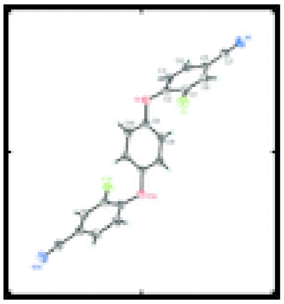

Fig. 1. Molecular structure of title compound with the atomic labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

Fig. 2. A partial packing diagram of title compound. Hydrogen bonds are shown as dashed lines. [Symmetry code: (i) -*x*, -*y*, -*z* + 2].

3,3'-Difluoro-4,4'-(*p*-phenylenedioxy)dibenzonitrile

Crystal data

$C_{20}H_{10}F_2N_2O_2$	$Z = 1$
$M_r = 348.30$	$F_{000} = 178$
Triclinic, $P\bar{1}$	$D_x = 1.469$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 6.980$ (1) Å	Cell parameters from 1140 reflections
$b = 7.615$ (1) Å	$\theta = 5.2$–54.9°
$c = 8.294$ (1) Å	$\mu = 0.11$ mm^{-1}
$\alpha = 106.376$ (3)°	$T = 293$ K
$\beta = 93.698$ (3)°	Prismatic, colorless
$\gamma = 109.085$ (3)°	0.42 × 0.37 × 0.32 mm
$V = 393.7$ (1) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	1529 independent reflections
Radiation source: fine-focus sealed tube	1259 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.065$
$T = 293$ K	$\theta_{max} = 26.0$°
φ and ω scans	$\theta_{min} = 2.6$°
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -8 \rightarrow 7$
$T_{min} = 0.782$, $T_{max} = 1.000$	$k = -9 \rightarrow 8$
2165 measured reflections	$l = -10 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.047$

$wR(F^2) = 0.137$

$S = 1.07$

1529 reflections

119 parameters

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

$w = 1/[\sigma^2(F_o^2) + (0.0836P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.19$ e Å^{-3}

$\Delta\rho_{min} = -0.21$ e Å^{-3}

Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.027 (6)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.31400 (15)	0.41880 (17)	0.64633 (14)	0.0546 (4)
N1	0.2645 (2)	0.0341 (2)	1.2835 (2)	0.0706 (5)
F1	0.21076 (16)	0.59370 (13)	0.94706 (13)	0.0663 (4)
C1	0.2380 (2)	0.4219 (2)	0.9251 (2)	0.0463 (4)
C2	0.2823 (2)	0.3307 (2)	0.77070 (18)	0.0443 (4)
C3	0.3137 (2)	0.1573 (2)	0.7484 (2)	0.0520 (4)
H3	0.3429	0.0941	0.6448	0.062*
C4	0.3023 (2)	0.0761 (2)	0.8780 (2)	0.0524 (4)
H4	0.3229	−0.0419	0.8619	0.063*
C5	0.2601 (2)	0.1714 (2)	1.03224 (18)	0.0460 (4)
C6	0.2256 (2)	0.3453 (2)	1.05636 (19)	0.0483 (4)
H6	0.1949	0.4084	1.1592	0.058*
C7	0.2590 (2)	0.0934 (2)	1.1718 (2)	0.0535 (4)
C8	0.1518 (2)	0.4580 (2)	0.57622 (16)	0.0424 (4)
C9	−0.0514 (2)	0.3477 (2)	0.56761 (18)	0.0478 (4)
H9	−0.0856	0.2450	0.6129	0.057*

C10	0.2042 (2)	0.6087 (2)	0.50919 (18)	0.0472 (4)
H10	0.3420	0.6816	0.5152	0.057*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0475 (6)	0.0766 (8)	0.0542 (7)	0.0254 (5)	0.0116 (5)	0.0384 (6)
N1	0.0775 (11)	0.0788 (11)	0.0601 (9)	0.0206 (8)	0.0085 (8)	0.0390 (8)
F1	0.0825 (8)	0.0573 (7)	0.0671 (7)	0.0349 (5)	0.0118 (5)	0.0209 (5)
C1	0.0428 (8)	0.0463 (8)	0.0503 (9)	0.0176 (6)	0.0023 (6)	0.0158 (7)
C2	0.0385 (7)	0.0561 (9)	0.0407 (8)	0.0150 (6)	0.0028 (6)	0.0222 (7)
C3	0.0585 (9)	0.0626 (10)	0.0412 (8)	0.0290 (8)	0.0095 (7)	0.0172 (7)
C4	0.0578 (9)	0.0548 (9)	0.0510 (9)	0.0258 (7)	0.0070 (7)	0.0206 (7)
C5	0.0397 (8)	0.0560 (9)	0.0429 (8)	0.0144 (6)	0.0018 (6)	0.0211 (7)
C6	0.0451 (8)	0.0586 (9)	0.0389 (8)	0.0177 (7)	0.0049 (6)	0.0141 (7)
C7	0.0481 (9)	0.0621 (10)	0.0496 (9)	0.0149 (7)	0.0036 (7)	0.0238 (8)
C8	0.0453 (8)	0.0489 (8)	0.0338 (7)	0.0167 (6)	0.0029 (6)	0.0160 (6)
C9	0.0496 (9)	0.0461 (8)	0.0472 (9)	0.0093 (6)	0.0032 (6)	0.0247 (7)
C10	0.0401 (8)	0.0512 (9)	0.0454 (8)	0.0058 (6)	0.0033 (6)	0.0217 (7)

Geometric parameters (Å, °)

O1—C2	1.3731 (16)	C4—H4	0.9300
O1—C8	1.3943 (16)	C5—C6	1.385 (2)
N1—C7	1.143 (2)	C5—C7	1.442 (2)
F1—C1	1.3469 (17)	C6—H6	0.9300
C1—C6	1.368 (2)	C8—C10	1.3678 (19)
C1—C2	1.381 (2)	C8—C9	1.377 (2)
C2—C3	1.372 (2)	C9—C10^{i}	1.385 (2)
C3—C4	1.378 (2)	C9—H9	0.9300
C3—H3	0.9300	C10—C9^{i}	1.385 (2)
C4—C5	1.384 (2)	C10—H10	0.9300
C2—O1—C8	118.23 (11)	C6—C5—C7	119.29 (14)
F1—C1—C6	119.41 (14)	C1—C6—C5	118.30 (14)
F1—C1—C2	118.49 (13)	C1—C6—H6	120.8
C6—C1—C2	122.08 (14)	C5—C6—H6	120.8
C3—C2—O1	119.59 (13)	N1—C7—C5	177.94 (17)
C3—C2—C1	118.81 (13)	C10—C8—C9	120.96 (13)
O1—C2—C1	121.32 (13)	C10—C8—O1	116.41 (12)
C2—C3—C4	120.60 (14)	C9—C8—O1	122.58 (12)
C2—C3—H3	119.7	C8—C9—C10^{i}	119.29 (13)
C4—C3—H3	119.7	C8—C9—H9	120.4
C3—C4—C5	119.53 (15)	C10^{i}—C9—H9	120.4
C3—C4—H4	120.2	C8—C10—C9^{i}	119.75 (13)
C5—C4—H4	120.2	C8—C10—H10	120.1
C4—C5—C6	120.67 (13)	C9^{i}—C10—H10	120.1
C4—C5—C7	120.00 (15)		

C8—O1—C2—C3	123.31 (15)	C2—C1—C6—C5	0.4 (2)
C8—O1—C2—C1	−62.84 (18)	C4—C5—C6—C1	−1.1 (2)
F1—C1—C2—C3	178.88 (13)	C7—C5—C6—C1	176.65 (13)
C6—C1—C2—C3	0.4 (2)	C4—C5—C7—N1	80 (5)
F1—C1—C2—O1	5.0 (2)	C6—C5—C7—N1	−98 (5)
C6—C1—C2—O1	−173.52 (13)	C2—O1—C8—C10	154.42 (13)
O1—C2—C3—C4	173.66 (13)	C2—O1—C8—C9	−28.1 (2)
C1—C2—C3—C4	−0.3 (2)	C10—C8—C9—C10^{i}	−0.4 (2)
C2—C3—C4—C5	−0.4 (2)	O1—C8—C9—C10^{i}	−177.78 (13)
C3—C4—C5—C6	1.2 (2)	C9—C8—C10—C9^{i}	0.4 (2)
C3—C4—C5—C7	−176.60 (14)	O1—C8—C10—C9^{i}	177.93 (12)
F1—C1—C6—C5	−178.13 (13)		

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C9—H9···N1^{ii}	0.93	2.50	3.410 (2)	166

Symmetry codes: (ii) −*x*, −*y*, −*z*+2.

Fig. 1

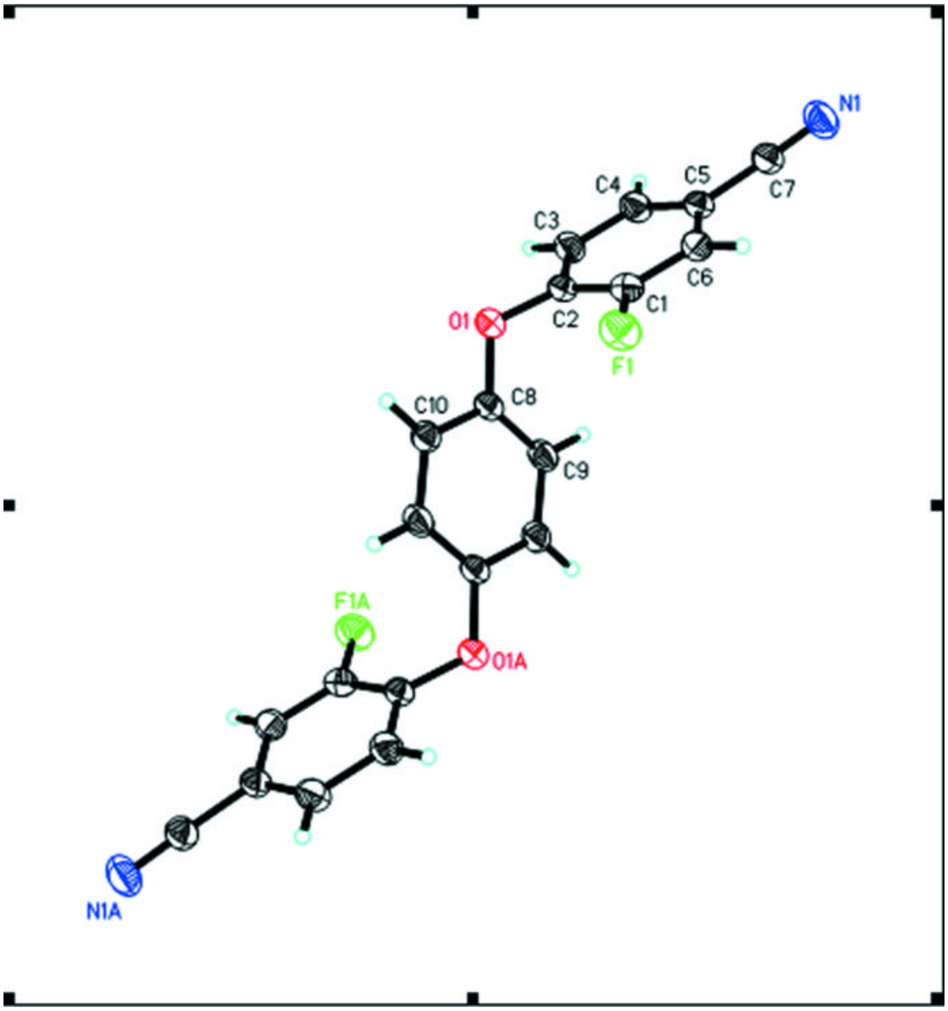

Fig. 2