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N,*N*′-[4,4′-Methylenebis(4,1-phenylene)]bis(2,6-difluorobenzamide)

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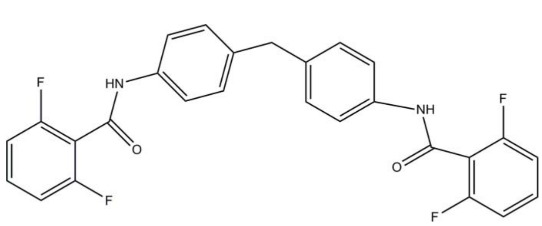
Key indicators: single-crystal X-ray study; *T* = 100 K; mean σ(C–C) = 0.001 Å; *R* factor = 0.040; *wR* factor = 0.114; data-to-parameter ratio = 23.7.

The complete molecule of the title compound, $C_{27}H_{18}F_4N_2O_2$, is generated by crystallographic twofold symmetry, with one C atom lying on the rotation axis. The dihedral angle between fluoro-substituted phenyl ring and the adjacent benzene ring is 10.37 (5)°. In the crystal, molecules are connected by N—H···O and C—H···F hydrogen bonds, resulting in supramolecular chains propagating along the *c* direction.

Related literature

For applications of benzamide derivatives, see: Ashwood *et al.* (1990); Kees *et al.* (1989); Ragavan *et al.* (2010); Carmellino *et al.* (1994); Rauko *et al.* (2001). For a related structure, see: Cronin *et al.* (2000). For the stability of the temperature controller used in the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_{27}H_{18}F_4N_2O_2$
M_r = 478.43
Monoclinic, *C*2/*c*
a = 42.0478 (10) Å
b = 5.2980 (1) Å
c = 9.5643 (2) Å
β = 92.522 (2)°
V = 2128.57 (8) Å^3
Z = 4
Mo *K*α radiation
μ = 0.12 mm^{-1}
T = 100 K
0.48 × 0.38 × 0.05 mm

Data collection

Bruker APEXII DUO CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.946, T_{max} = 0.994
26445 measured reflections
3871 independent reflections
3172 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.114
S = 1.06
3871 reflections
163 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.43 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*
N1—H1*N*1···O1^{i}	0.841 (15)	2.078 (15)	2.8811 (11)	159.4 (14)
C9—H9*A*···F1^{ii}	0.95	2.41	3.2318 (11)	145

Symmetry codes: (i) $x, -y+2, z-\frac{1}{2}$; (ii) $x, -y+2, z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5924).

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supplementary materials

Acta Cryst. (2011). E**67**, o1832 [doi:10.1107/S1600536811024524]

N,*N*'-[4,4'-Methylenebis(4,1-phenylene)]bis(2,6-difluorobenzamide)

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Comment

A number of benzamide derivatives were reported to possess anti-hypertensive (Ashwood *et al.*, 1990), anti-diabetic (Kees *et al.*, 1989), anti-bacterial (Ragavan *et al.*, 2010), anti-fungal (Carmellino *et al.*, 1994) and anti-cancer (Rauko *et al.*, 2001) activities. As a part of our study on the synthesis of new fluorine- containing compounds with possible biological activities, we report here the crystal structure of the title compound, (I).

The asymmetric unit of the title compound, (Fig. 1), consists of a half molecule of *N,N'*-(4,4'-methylenebis(4,1-phenylene))bis (2,6-difluorobenzamide), which has a twofold symmetry and it adopts an *E, E* conformation. The dihedral angle between fluoro- substituted phenyl (C1–C6) rings and benzene (C8–13) ring is 10.37 (5)°. All bond lengths and angles are comparable to values observed in a closely related benzamide structure (Cronin *et al.*, 2000).

In the crystal structure (Fig. 2), the adjacent molecules are connected *via* N1—H1N1..O1 and C9—H9A···F1 (Table 1) hydrogen bonds forming one-dimensional supramolecular chains along the *c*-axis.

Experimental

In a round bottom flask, 25ml of tetrahydrofuran (THF) was mixed with 4,4'-diaminodiphenylmethane (0.01 mol,2 g) with stirring. Drops of 2,6-Difluorobenzylchloride (0.02 mol, 3.4 g) which was dissolved in THF was then added. The mixture was refluxed for 30 min and the yellow precipitate formed was filtered and washed with alkaline water, then with toluene and further washed with dilute hydrochloric acid. The precipate was dissolved in methanol at room temperature yielding colourless plates of (I).

Refinement

Atom H1N1 was located from a difference Fourier maps and refined freely [N–H = 0.842 (15) Å]. The remaining H atoms were positioned geometrically [C–H = 0.95–0.9601 Å] and were refined using a riding model, with U_{iso}(H) = 1.2 U_{eq}(C). The highest residual electron density peak is located at 0.61 Å from C6 and the deepest hole 0.64 Å located at from C1.

Figures

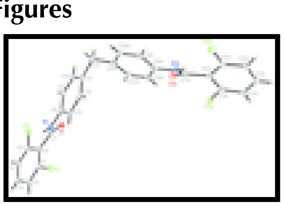


Fig. 1. The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids.

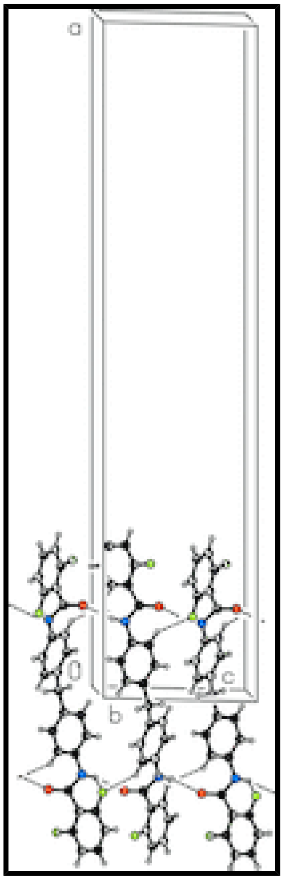


Fig. 2. A view of a one-dimensional supramolecular chain along the *c*-axis

N-{4-[4-(2,6-difluorobenzamido)benzyl]phenyl}-2,6-difluorobenzamide

Crystal data

$C_{27}H_{18}F_4N_2O_2$	$F(000) = 984$
$M_r = 478.43$	$D_x = 1.493$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 8910 reflections
$a = 42.0478$ (10) Å	$\theta = 2.9$–32.5°
$b = 5.2980$ (1) Å	$\mu = 0.12$ mm^{-1}
$c = 9.5643$ (2) Å	$T = 100$ K
$\beta = 92.522$ (2)°	Plate, colourless
$V = 2128.57$ (8) Å^3	0.48 × 0.38 × 0.05 mm
$Z = 4$	

Data collection

Bruker APEXII DUO CCD diffractometer	3871 independent reflections
Radiation source: fine-focus sealed tube	3172 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.031$
φ and ω scans	$\theta_{max} = 32.6°$, $\theta_{min} = 1.9°$

Absorption correction: multi-scan (*SADABS*; Bruker, 2009)	$h = -63 \rightarrow 63$
$T_{min} = 0.946$, $T_{max} = 0.994$	$k = -7 \rightarrow 7$
26445 measured reflections	$l = -14 \rightarrow 14$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.040$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.114$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.06$	$w = 1/[\sigma^2(F_o^2) + (0.0569P)^2 + 1.3081P]$ where $P = (F_o^2 + 2F_c^2)/3$
3871 reflections	$(\Delta/\sigma)_{max} < 0.001$
163 parameters	$\Delta\rho_{max} = 0.43$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1) K.

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'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 $F^2 > 2\sigma(F^2)$ 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}
F1	0.131371 (14)	0.52803 (12)	0.04350 (6)	0.02016 (14)
F2	0.201443 (15)	1.06377 (13)	0.30050 (7)	0.02303 (15)
O1	0.136883 (18)	0.97437 (15)	0.36217 (7)	0.02009 (16)
N1	0.113987 (19)	1.00956 (16)	0.14156 (8)	0.01541 (16)
C1	0.16118 (2)	0.60034 (18)	0.08307 (9)	0.01554 (17)
C2	0.18629 (3)	0.4631 (2)	0.03344 (10)	0.01989 (19)
H2A	0.1826	0.3219	−0.0264	0.024*
C3	0.21695 (2)	0.5379 (2)	0.07365 (11)	0.0223 (2)
H3A	0.2346	0.4482	0.0398	0.027*
C4	0.22225 (2)	0.7422 (2)	0.16280 (11)	0.0207 (2)
H4A	0.2433	0.7941	0.1895	0.025*

C5	0.19617 (2)	0.86802 (19)	0.21155 (10)	0.01656 (18)
C6	0.16482 (2)	0.80494 (17)	0.17353 (9)	0.01396 (16)
C7	0.13715 (2)	0.93935 (17)	0.23513 (9)	0.01430 (17)
C8	0.08569 (2)	1.13880 (18)	0.17534 (9)	0.01475 (17)
C9	0.08638 (2)	1.33660 (19)	0.27115 (10)	0.01791 (18)
H9A	0.1058	1.3826	0.3189	0.021*
C10	0.05852 (2)	1.46660 (19)	0.29678 (10)	0.01816 (18)
H10A	0.0591	1.6004	0.3630	0.022*
C11	0.02971 (2)	1.40427 (18)	0.22709 (10)	0.01674 (18)
C12	0.02942 (2)	1.20383 (19)	0.13243 (11)	0.01962 (19)
H12A	0.0100	1.1570	0.0852	0.024*
C13	0.05708 (2)	1.07101 (19)	0.10588 (10)	0.01794 (18)
H13A	0.0565	0.9352	0.0409	0.022*
C14	0.0000	1.5573 (3)	0.2500	0.0213 (3)
H14A	−0.0040	1.6645	0.1702	0.026*
H1N1	0.1173 (3)	0.984 (3)	0.0566 (16)	0.023 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0186 (3)	0.0209 (3)	0.0208 (3)	−0.0022 (2)	−0.0022 (2)	−0.0022 (2)
F2	0.0207 (3)	0.0257 (3)	0.0225 (3)	−0.0035 (2)	−0.0013 (2)	−0.0072 (2)
O1	0.0220 (3)	0.0272 (4)	0.0112 (3)	0.0051 (3)	0.0022 (2)	0.0005 (3)
N1	0.0144 (3)	0.0210 (4)	0.0110 (3)	0.0032 (3)	0.0022 (3)	−0.0003 (3)
C1	0.0159 (4)	0.0180 (4)	0.0127 (4)	0.0003 (3)	0.0000 (3)	0.0020 (3)
C2	0.0249 (5)	0.0197 (5)	0.0151 (4)	0.0056 (4)	0.0019 (3)	−0.0017 (3)
C3	0.0201 (4)	0.0269 (5)	0.0202 (4)	0.0078 (4)	0.0043 (4)	0.0006 (4)
C4	0.0148 (4)	0.0268 (5)	0.0205 (4)	0.0026 (3)	0.0012 (3)	0.0017 (4)
C5	0.0168 (4)	0.0189 (4)	0.0139 (4)	−0.0001 (3)	−0.0001 (3)	−0.0001 (3)
C6	0.0146 (4)	0.0161 (4)	0.0112 (3)	0.0008 (3)	0.0011 (3)	0.0009 (3)
C7	0.0150 (4)	0.0151 (4)	0.0130 (4)	0.0003 (3)	0.0022 (3)	0.0010 (3)
C8	0.0137 (4)	0.0176 (4)	0.0132 (4)	0.0012 (3)	0.0024 (3)	0.0011 (3)
C9	0.0151 (4)	0.0220 (4)	0.0166 (4)	0.0002 (3)	0.0003 (3)	−0.0031 (3)
C10	0.0166 (4)	0.0194 (4)	0.0186 (4)	0.0005 (3)	0.0023 (3)	−0.0036 (3)
C11	0.0139 (4)	0.0158 (4)	0.0208 (4)	0.0000 (3)	0.0042 (3)	0.0014 (3)
C12	0.0145 (4)	0.0198 (4)	0.0244 (5)	−0.0008 (3)	−0.0001 (3)	−0.0023 (4)
C13	0.0166 (4)	0.0185 (4)	0.0187 (4)	0.0000 (3)	0.0004 (3)	−0.0032 (3)
C14	0.0140 (6)	0.0159 (6)	0.0343 (8)	0.000	0.0044 (5)	0.000

Geometric parameters (Å, °)

F1—C1	1.3489 (11)	C6—C7	1.5058 (13)
F2—C5	1.3534 (11)	C8—C9	1.3916 (13)
O1—C7	1.2297 (11)	C8—C13	1.3954 (13)
N1—C7	1.3459 (12)	C9—C10	1.3901 (13)
N1—C8	1.4222 (12)	C9—H9A	0.9500
N1—H1N1	0.842 (15)	C10—C11	1.3963 (13)
C1—C2	1.3828 (13)	C10—H10A	0.9500
C1—C6	1.3911 (13)	C11—C12	1.3952 (14)

C2—C3	1.3870 (15)	C11—C14	1.5131 (12)
C2—H2A	0.9500	C12—C13	1.3922 (13)
C3—C4	1.3899 (15)	C12—H12A	0.9500
C3—H3A	0.9500	C13—H13A	0.9500
C4—C5	1.3815 (13)	C14—C11^{i}	1.5131 (12)
C4—H4A	0.9500	C14—H14A	0.9601
C5—C6	1.3929 (12)		

C7—N1—C8	124.77 (8)	N1—C7—C6	114.80 (8)
C7—N1—H1N1	116.8 (10)	C9—C8—C13	119.99 (9)
C8—N1—H1N1	118.2 (10)	C9—C8—N1	121.25 (8)
F1—C1—C2	117.93 (9)	C13—C8—N1	118.70 (8)
F1—C1—C6	118.10 (8)	C10—C9—C8	119.73 (9)
C2—C1—C6	123.96 (9)	C10—C9—H9A	120.1
C1—C2—C3	117.96 (9)	C8—C9—H9A	120.1
C1—C2—H2A	121.0	C9—C10—C11	121.26 (9)
C3—C2—H2A	121.0	C9—C10—H10A	119.4
C2—C3—C4	120.98 (9)	C11—C10—H10A	119.4
C2—C3—H3A	119.5	C12—C11—C10	118.18 (9)
C4—C3—H3A	119.5	C12—C11—C14	121.20 (8)
C5—C4—C3	118.33 (9)	C10—C11—C14	120.57 (8)
C5—C4—H4A	120.8	C13—C12—C11	121.31 (9)
C3—C4—H4A	120.8	C13—C12—H12A	119.3
F2—C5—C4	118.12 (8)	C11—C12—H12A	119.3
F2—C5—C6	118.35 (8)	C12—C13—C8	119.51 (9)
C4—C5—C6	123.53 (9)	C12—C13—H13A	120.2
C1—C6—C5	115.22 (8)	C8—C13—H13A	120.2
C1—C6—C7	123.11 (8)	C11—C14—C11^{i}	115.22 (11)
C5—C6—C7	121.53 (8)	C11—C14—H14A	108.5
O1—C7—N1	125.27 (9)	C11^{i}—C14—H14A	108.4
O1—C7—C6	119.92 (8)		

F1—C1—C2—C3	179.76 (9)	C5—C6—C7—O1	−46.84 (13)
C6—C1—C2—C3	−1.62 (15)	C1—C6—C7—N1	−50.49 (12)
C1—C2—C3—C4	0.95 (16)	C5—C6—C7—N1	133.95 (9)
C2—C3—C4—C5	0.57 (16)	C7—N1—C8—C9	42.34 (14)
C3—C4—C5—F2	178.73 (9)	C7—N1—C8—C13	−140.39 (10)
C3—C4—C5—C6	−1.60 (15)	C13—C8—C9—C10	−0.27 (15)
F1—C1—C6—C5	179.29 (8)	N1—C8—C9—C10	176.96 (9)
C2—C1—C6—C5	0.68 (14)	C8—C9—C10—C11	−0.61 (15)
F1—C1—C6—C7	3.47 (13)	C9—C10—C11—C12	1.22 (15)
C2—C1—C6—C7	−175.14 (9)	C9—C10—C11—C14	−176.46 (9)
F2—C5—C6—C1	−179.35 (8)	C10—C11—C12—C13	−0.98 (15)
C4—C5—C6—C1	0.98 (14)	C14—C11—C12—C13	176.68 (9)
F2—C5—C6—C7	−3.46 (13)	C11—C12—C13—C8	0.14 (15)
C4—C5—C6—C7	176.87 (9)	C9—C8—C13—C12	0.50 (15)
C8—N1—C7—O1	0.91 (16)	N1—C8—C13—C12	−176.80 (9)
C8—N1—C7—C6	−179.94 (8)	C12—C11—C14—C11^{i}	47.96 (8)
C1—C6—C7—O1	128.72 (10)	C10—C11—C14—C11^{i}	−134.43 (10)

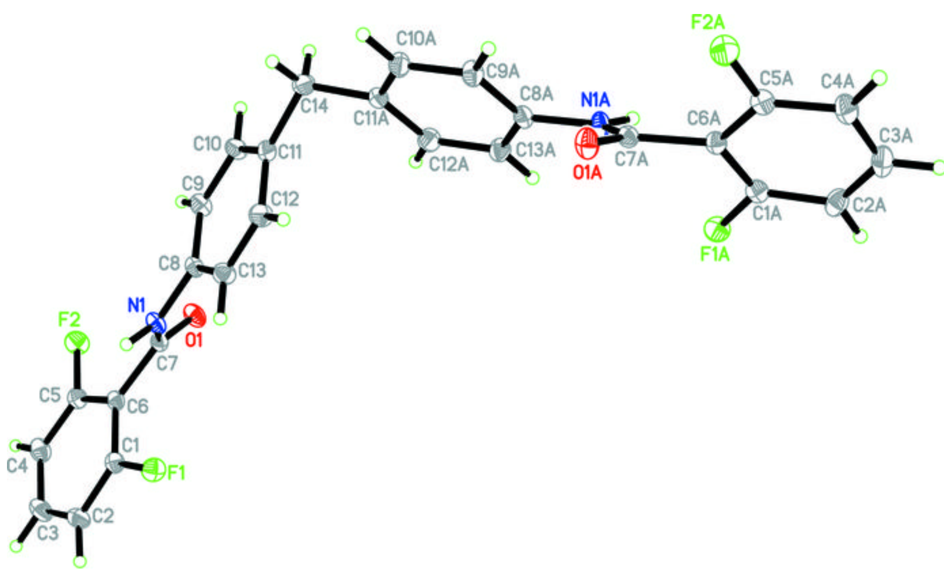
Symmetry codes: (i) −*x*, *y*, −*z*+1/2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···O1^{ii}	0.841 (15)	2.078 (15)	2.8811 (11)	159.4 (14)
C9—H9A···F1^{iii}	0.95	2.41	3.2318 (11)	145

Symmetry codes: (ii) *x*, −*y*+2, *z*−1/2; (iii) *x*, −*y*+2, *z*+1/2.

Fig. 1


F2A
C10A
C9A
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C4A
C14
C8A
N1A
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C11A
C3A
C10
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C13A
O1A
C7A
C9
C1A
C2A
C12
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C6
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Fig. 2