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4-Methoxy-2-nitro-4′-(trifluoromethyl)-biphenyl

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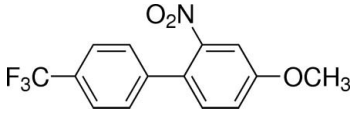
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; disorder in main residue; R factor = 0.050; wR factor = 0.161; data-to-parameter ratio = 13.3.

The title compound, $C_{14}H_{10}F_3NO_3$, was prepared by a palladium-catalysed Suzuki–Miyaura coupling reaction. The dihedral angle between the nitro group and its parent benzene ring is 66.85 (19)° while the dihedral angle between the two benzene rings is 49.98 (9)°. The CF_3 group is disordered over two sets of sites with occupancies of 0.457 (8) and 0.543 (8).

Related literature

For general background to the synthesis and properties of the title compound, see: Suzuki (1999); Razler *et al.* (2009). For the biological activity of biphenyl derivatives, see: Kimpe *et al.* (1996).



Experimental

Crystal data

$C_{14}H_{10}F_3NO_3$ M_r = 297.23
Monoclinic, $P2_1/c$
a = 8.1956 (13) Å
b = 20.777 (3) Å
c = 7.9715 (12) Å
β = 104.240 (2)°
V = 1315.7 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 K
0.26 × 0.24 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.966, T_{max} = 0.974
10512 measured reflections
3235 independent reflections
1910 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.161
S = 1.05
3235 reflections
243 parameters
36 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); 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: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FF2027).

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supporting information

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4-Methoxy-2-nitro-4′-(trifluoromethyl)biphenyl

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S1. Experimental

To a solution of 4-bromo-trifluoromethylphenyl (5 mmol) and 4-methoxy-2-nitro-phenylboronic acid (6 mmol) in 20 ml water and 20 ml methanol was added $Pd(OAc)_2$ (5 mmol) and K_2CO_3 (10 mmol). After stirring the reaction mixture for 12 h at room temperature, the aqueous phases were extracted with 100 ml ethyl acetate. The organic extracts were washed with 200 ml saturated aqueous sodium chloride, dried over sodium sulfate, filtered, and concentrated under reduced pressure. The resulting crude material was purified *via* silica gel chromatography (5% ethyl acetate/hexane) to afford a translucent solid in a yield of 80%. Crystals suitable for single-crystal X-ray diffraction were obtained by recrystallization from methanol at room temperature in a total yield of 32%. Analysis found: C 56.6, H 3.3, N 4.6%; $C_{14}H_{10}F_3NO_3$ requires: C 56.6, H 3.4, N 4.7%. $_1$H NMR (400 MHz, $CDCl_3$) 7.66 (d, J = 8.1 Hz, 2H), 7.45 (d, J = 2.6 Hz, 1H), 7.40 (d, J = 8.0 Hz, 2H), 7.32 (d, J = 8.5 Hz, 1H), 7.19 (dd, J = 8.6, 2.6 Hz, 1H), 3.92 (s, 3H).

S2. Refinement

All H-atoms were positioned geometrically and included in the refinement in the riding-model approximation, with U_{iso}(H)= 1.5Ueq(methyl C) and 1.2Ueq(aromatic C). The –CF_3 group is disordered over two sites with occupancies of 0.457 (8) and 0.543 (8). For this fragment, some anisotropic displacement ellipsoids were rather elongated which led us to use the ISOR restraints (Sheldrick, 2008).

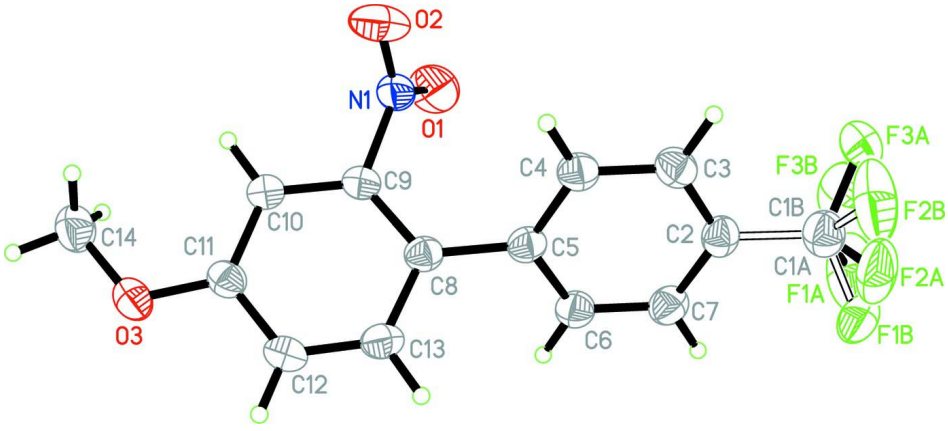

Figure 1

The structure of (I) with 50% probability displacement ellipsoids for non-hydrogen atoms showing the disordered –CF_3 group.

4-Methoxy-2-nitro-4′-(trifluoromethyl)biphenyl

Crystal data

$C_{14}H_{10}F_3NO_3$
$M_r = 297.23$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.1956$ (13) Å
$b = 20.777$ (3) Å
$c = 7.9715$ (12) Å
$\beta = 104.240$ (2)°
$V = 1315.7$ (3) Å^3
$Z = 4$
$F(000) = 608$
$D_x = 1.501$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1880 reflections
$\theta = 2.8$–22.6°
$\mu = 0.13$ mm^{-1}
$T = 293$ K
Block, colorless
0.26 × 0.24 × 0.20 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.966$, $T_{max} = 0.974$
10512 measured reflections
3235 independent reflections
1910 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.8°$
$h = -10 \rightarrow 10$
$k = -27 \rightarrow 27$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.161$
$S = 1.05$
3235 reflections
243 parameters
36 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0741P)^2 + 0.1504P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL*, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.026 (4)

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}	Occ. (<1)
C1A	0.6447 (4)	0.19880 (14)	0.4301 (4)	0.0705 (7)	0.46
F1A	0.6131 (8)	0.1395 (3)	0.3972 (18)	0.147 (3)	0.46
F2A	0.7487 (9)	0.2001 (4)	0.5832 (6)	0.108 (2)	0.46

F3A	0.7514 (8)	0.2162 (4)	0.3299 (10)	0.1111 (16)	0.46
C1B	0.6447 (4)	0.19880 (14)	0.4301 (4)	0.0705 (7)	0.54
F1B	0.6331 (8)	0.1469 (3)	0.5270 (8)	0.1152 (18)	0.54
F2B	0.7864 (6)	0.2248 (3)	0.4985 (14)	0.142 (2)	0.54
F3B	0.6443 (9)	0.1723 (3)	0.2844 (5)	0.1143 (16)	0.54
O3	−0.3255 (2)	0.48543 (8)	0.4265 (2)	0.0667 (5)	
N1	0.0999 (2)	0.42470 (11)	0.1294 (2)	0.0604 (5)	
C2	0.4994 (3)	0.24308 (11)	0.4159 (3)	0.0549 (6)	
C3	0.5169 (3)	0.30787 (12)	0.3869 (3)	0.0581 (6)	
C4	0.3843 (3)	0.34936 (11)	0.3798 (3)	0.0551 (6)	
C5	0.2308 (3)	0.32692 (10)	0.4005 (3)	0.0496 (5)	
C6	0.2145 (3)	0.26131 (11)	0.4274 (3)	0.0573 (6)	
C7	0.3473 (3)	0.21981 (12)	0.4348 (3)	0.0606 (6)	
C8	0.0882 (3)	0.37089 (10)	0.4025 (3)	0.0485 (5)	
C9	0.0257 (3)	0.41719 (10)	0.2790 (3)	0.0484 (5)	
C10	−0.1091 (3)	0.45702 (10)	0.2791 (3)	0.0504 (5)	
C11	−0.1881 (3)	0.45081 (10)	0.4134 (3)	0.0512 (5)	
C12	−0.1259 (3)	0.40693 (11)	0.5441 (3)	0.0574 (6)	
H12	−0.1751	0.4039	0.6372	0.069*	
C13	0.0072 (3)	0.36797 (11)	0.5379 (3)	0.0568 (6)	
C14	−0.4039 (3)	0.52531 (14)	0.2844 (4)	0.0754 (8)	
H14A	−0.3272	0.5585	0.2700	0.113*	
H14B	−0.5032	0.5445	0.3064	0.113*	
H14C	−0.4343	0.4998	0.1810	0.113*	
H10	−0.146 (3)	0.4878 (10)	0.188 (3)	0.053 (6)*	
H4	0.401 (3)	0.3937 (11)	0.364 (3)	0.064 (7)*	
H11	0.044 (3)	0.3381 (11)	0.630 (3)	0.063 (6)*	
H6	0.112 (3)	0.2454 (11)	0.442 (3)	0.062 (6)*	
H7	0.332 (3)	0.1735 (12)	0.454 (3)	0.067 (7)*	
H3	0.622 (3)	0.3236 (11)	0.371 (3)	0.068 (7)*	
O2	0.1707 (3)	0.47594 (11)	0.1180 (3)	0.0972 (7)	
O1	0.0856 (3)	0.38237 (10)	0.0266 (3)	0.1025 (8)	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1A	0.0697 (18)	0.0762 (18)	0.0648 (17)	0.0077 (14)	0.0150 (13)	−0.0032 (14)
F1A	0.114 (4)	0.079 (3)	0.239 (7)	0.007 (3)	0.025 (6)	−0.054 (5)
F2A	0.099 (4)	0.151 (6)	0.063 (2)	0.062 (4)	−0.001 (2)	0.004 (3)
F3A	0.089 (3)	0.141 (4)	0.123 (4)	0.043 (3)	0.061 (3)	0.041 (3)
C1B	0.0697 (18)	0.0762 (18)	0.0648 (17)	0.0077 (14)	0.0150 (13)	−0.0032 (14)
F1B	0.126 (4)	0.116 (4)	0.115 (3)	0.066 (3)	0.053 (3)	0.055 (3)
F2B	0.065 (2)	0.122 (4)	0.221 (6)	0.017 (2)	0.002 (4)	−0.038 (4)
F3B	0.151 (4)	0.128 (3)	0.070 (2)	0.067 (3)	0.039 (2)	−0.007 (2)
O3	0.0589 (10)	0.0774 (11)	0.0701 (11)	0.0099 (8)	0.0280 (8)	0.0027 (8)
N1	0.0601 (12)	0.0727 (13)	0.0524 (11)	0.0114 (10)	0.0215 (9)	0.0146 (10)
C2	0.0553 (13)	0.0601 (13)	0.0466 (12)	0.0065 (11)	0.0076 (10)	−0.0014 (9)
C3	0.0517 (13)	0.0662 (15)	0.0568 (13)	−0.0055 (11)	0.0139 (10)	−0.0024 (11)

C4	0.0562 (14)	0.0512 (13)	0.0586 (13)	−0.0068 (11)	0.0152 (10)	0.0017 (10)
C5	0.0518 (12)	0.0518 (12)	0.0436 (11)	−0.0029 (10)	0.0085 (9)	0.0027 (9)
C6	0.0535 (14)	0.0536 (13)	0.0655 (14)	−0.0051 (11)	0.0161 (11)	0.0067 (10)
C7	0.0651 (15)	0.0524 (13)	0.0637 (14)	0.0005 (12)	0.0148 (11)	0.0058 (11)
C8	0.0477 (12)	0.0475 (11)	0.0501 (12)	−0.0066 (9)	0.0115 (9)	0.0023 (9)
C9	0.0518 (12)	0.0519 (11)	0.0438 (11)	−0.0031 (9)	0.0166 (9)	0.0019 (9)
C10	0.0531 (12)	0.0499 (11)	0.0501 (12)	0.0002 (10)	0.0161 (10)	0.0056 (10)
C11	0.0489 (12)	0.0529 (12)	0.0545 (12)	−0.0062 (10)	0.0176 (10)	−0.0032 (10)
C12	0.0590 (14)	0.0657 (14)	0.0529 (12)	−0.0066 (11)	0.0243 (10)	0.0026 (10)
C13	0.0611 (14)	0.0604 (13)	0.0492 (12)	−0.0040 (11)	0.0144 (10)	0.0104 (10)
C14	0.0611 (16)	0.0862 (18)	0.0803 (18)	0.0158 (14)	0.0201 (13)	0.0066 (14)
O2	0.1010 (15)	0.1191 (17)	0.0805 (14)	−0.0256 (13)	0.0393 (12)	0.0224 (12)
O1	0.149 (2)	0.0977 (15)	0.0789 (13)	0.0180 (14)	0.0628 (14)	−0.0116 (11)

Geometric parameters (Å, º)

C1A—F1A	1.273 (6)	C5—C8	1.487 (3)
C1A—F2A	1.306 (6)	C6—C7	1.378 (3)
C1A—F3A	1.370 (5)	C6—H6	0.93 (2)
C1A—C2	1.487 (3)	C7—H7	0.99 (2)
O3—C11	1.361 (3)	C8—C9	1.381 (3)
O3—C14	1.423 (3)	C8—C13	1.401 (3)
N1—O1	1.188 (3)	C9—C10	1.381 (3)
N1—O2	1.226 (3)	C10—C11	1.387 (3)
N1—C9	1.474 (2)	C10—H10	0.96 (2)
C2—C3	1.379 (3)	C11—C12	1.384 (3)
C2—C7	1.380 (3)	C12—C13	1.369 (3)
C3—C4	1.377 (3)	C12—H12	0.9300
C3—H3	0.96 (2)	C13—H11	0.95 (2)
C4—C5	1.389 (3)	C14—H14A	0.9600
C4—H4	0.94 (2)	C14—H14B	0.9600
C5—C6	1.391 (3)	C14—H14C	0.9600
F1A—C1A—F2A	105.4 (6)	C6—C7—H7	119.2 (14)
F1A—C1A—F3A	105.2 (5)	C2—C7—H7	120.7 (14)
F2A—C1A—F3A	100.1 (5)	C9—C8—C13	114.61 (19)
F1A—C1A—C2	117.7 (4)	C9—C8—C5	125.07 (17)
F2A—C1A—C2	112.6 (3)	C13—C8—C5	120.31 (19)
F3A—C1A—C2	114.0 (3)	C10—C9—C8	124.99 (18)
C11—O3—C14	117.82 (17)	C10—C9—N1	115.23 (18)
O1—N1—O2	124.0 (2)	C8—C9—N1	119.73 (18)
O1—N1—C9	119.3 (2)	C9—C10—C11	118.1 (2)
O2—N1—C9	116.6 (2)	C9—C10—H10	120.4 (12)
C3—C2—C7	119.6 (2)	C11—C10—H10	121.5 (12)
C3—C2—C1A	120.2 (2)	O3—C11—C12	116.66 (18)
C7—C2—C1A	120.2 (2)	O3—C11—C10	124.2 (2)
C4—C3—C2	120.3 (2)	C12—C11—C10	119.1 (2)
C4—C3—H3	120.5 (14)	C13—C12—C11	120.76 (19)

C2—C3—H3	119.2 (14)	C13—C12—H12	119.6
C3—C4—C5	120.9 (2)	C11—C12—H12	119.6
C3—C4—H4	118.5 (15)	C12—C13—C8	122.4 (2)
C5—C4—H4	120.6 (15)	C12—C13—H11	117.6 (14)
C4—C5—C6	118.1 (2)	C8—C13—H11	120.1 (14)
C4—C5—C8	122.17 (19)	O3—C14—H14A	109.5
C6—C5—C8	119.66 (19)	O3—C14—H14B	109.5
C7—C6—C5	121.0 (2)	H14A—C14—H14B	109.5
C7—C6—H6	119.8 (14)	O3—C14—H14C	109.5
C5—C6—H6	119.2 (14)	H14A—C14—H14C	109.5
C6—C7—C2	120.1 (2)	H14B—C14—H14C	109.5
F1A—C1A—C2—C3	−156.3 (8)	C13—C8—C9—C10	−2.3 (3)
F2A—C1A—C2—C3	80.8 (6)	C5—C8—C9—C10	178.5 (2)
F3A—C1A—C2—C3	−32.4 (6)	C13—C8—C9—N1	−179.6 (2)
F1A—C1A—C2—C7	24.9 (8)	C5—C8—C9—N1	1.2 (3)
F2A—C1A—C2—C7	−98.0 (6)	O1—N1—C9—C10	−111.7 (2)
F3A—C1A—C2—C7	148.7 (5)	O2—N1—C9—C10	66.8 (3)
C7—C2—C3—C4	1.1 (3)	O1—N1—C9—C8	65.8 (3)
C1A—C2—C3—C4	−177.7 (2)	O2—N1—C9—C8	−115.6 (2)
C2—C3—C4—C5	−0.4 (3)	C8—C9—C10—C11	0.5 (3)
C3—C4—C5—C6	−0.4 (3)	N1—C9—C10—C11	177.87 (19)
C3—C4—C5—C8	176.8 (2)	C14—O3—C11—C12	−172.8 (2)
C4—C5—C6—C7	0.5 (3)	C14—O3—C11—C10	7.2 (3)
C8—C5—C6—C7	−176.7 (2)	C9—C10—C11—O3	−177.8 (2)
C5—C6—C7—C2	0.2 (4)	C9—C10—C11—C12	2.2 (3)
C3—C2—C7—C6	−1.0 (3)	O3—C11—C12—C13	177.1 (2)
C1A—C2—C7—C6	177.8 (2)	C10—C11—C12—C13	−2.9 (3)
C4—C5—C8—C9	50.9 (3)	C11—C12—C13—C8	0.9 (4)
C6—C5—C8—C9	−131.9 (2)	C9—C8—C13—C12	1.6 (3)
C4—C5—C8—C13	−128.2 (2)	C5—C8—C13—C12	−179.2 (2)
C6—C5—C8—C13	48.9 (3)		