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4-Methoxy-3-(trifluoromethyl)aniline

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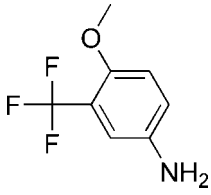
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.059; wR factor = 0.162; data-to-parameter ratio = 14.6.

In title compound, $C_8H_8F_3NO$, the methoxy group is inclined at 8.7 (4)° to the benzene ring plane. The crystal structure is stabilized by intermolecular N—H···F, N—H···N and C—H···F hydrogen-bonding interactions.

Related literature

The title compound is an intermediate in the synthesis of trifluoromethyl-containing phthalic acid diamides, which are effective pesticides. For the preparation, see: Feng & Li (2010). For the crystal structure of a closely related compound, see: Crampton *et al.* (2006).



Experimental

Crystal data

$C_8H_8F_3NO$
M_r = 191.15
Orthorhombic, *Pbca*
a = 5.4140 (11) Å
b = 14.880 (3) Å
c = 21.304 (4) Å
V = 1716.3 (6) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 293 K
0.10 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.986, T_{max} = 0.986
1722 measured reflections
1722 independent reflections
1389 reflections with $I > 2\sigma(I)$

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.162
S = 1.13
1722 reflections
118 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1C···F2^{i}	0.96	2.52	3.292 (3)	138
N—H0A···F1^{ii}	0.86	2.44	3.242 (2)	155
N—H0B···N^{iii}	0.86	2.47	3.245 (3)	150

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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*.

The author thanks the Center of Testing and Analysis, Nanjing University, for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2499).

References

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supporting information

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4-Methoxy-3-(trifluoromethyl)aniline

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S1. Comment

The tittle compound (Fig. 1) is used as an important intermediate in the synthesis of trifluoromethyl-containing phthalic acid compounds which are recognized as effective pesticides (Feng & Li, 2010). In the title molecule, the N, O and C8 atoms bonded to the central benzene ring (C2–C7) lie in its plane with methoxy group (O/C1) oriented at 8.7 (4) ° with respect to the benzene ring plane. There is an intramolecular interaction C6—H6A···F3 which stabilizes the molecular structure of the title compound. The crystal structure is stabilized by N—H0A···F1, N—H0B···N and C1—H1C···F2 intermolecular hydrogen bonding interactions. The bond distances and bond angles in the title compound agree with the corresponding bond distances and bond angles reported in a closely related compound (Crampton *et al.*, 2006).

S2. Experimental

The title compound was prepared by a method reported in the literature (Feng & Li, 2010)). A solution of 4-amino-2-(trifluoromethyl)phenol (2 g, 11.3 mmol) in dichloromethane (20 ml) was added slowly to a solution of sodium hydride (0.33 g, 13.6 nmol) in an ice bath. After stirring for 6 h iodomethane (4.8 g, 33.9 mmol) was added slowly in 1 h. After stirring for 48 h at room tempeature, the solvent was evaporated on a rotary evaporator yielding the title compound. Colorless blocks of the title compound were grown in ethanol (20 ml) by slow evaporation of the solvent at room temperature in about 7 days.

S3. Refinement

The H atoms were positioned geometrically and constrained to ride on their parent atoms, with N—H = 0.86 Å and C—H = 0.93 and 0.96 Å for aryl and alky H-atoms, respectively, with U_{iso}(H) = xU_{eq}(C/N), where x = 1.2 for aromatic and amino H, and x = 1.5 for aryl H atoms.

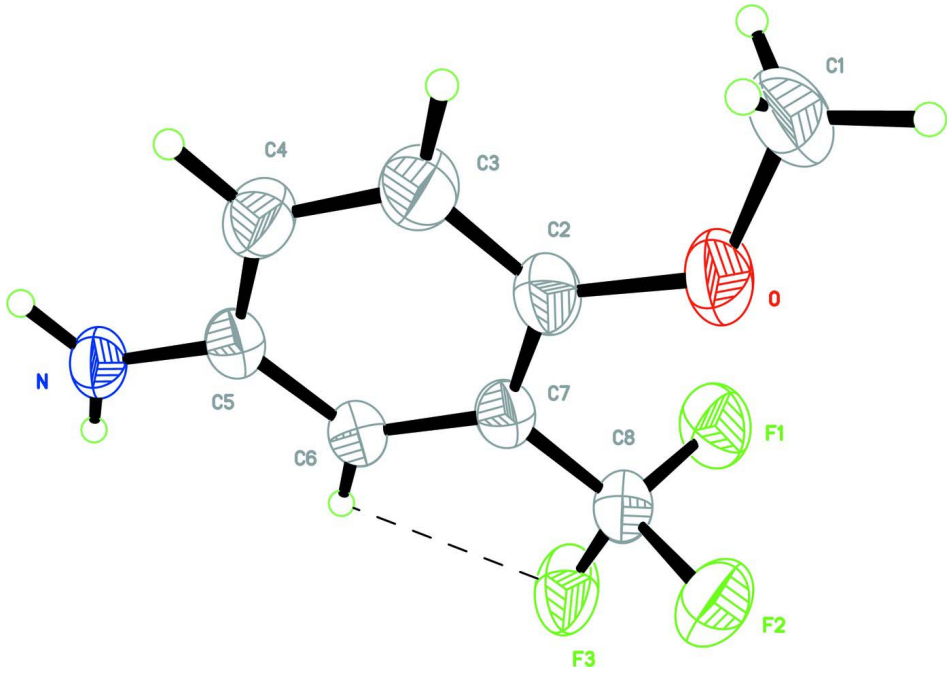

Figure 1

The molecular structure of the title molecule with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

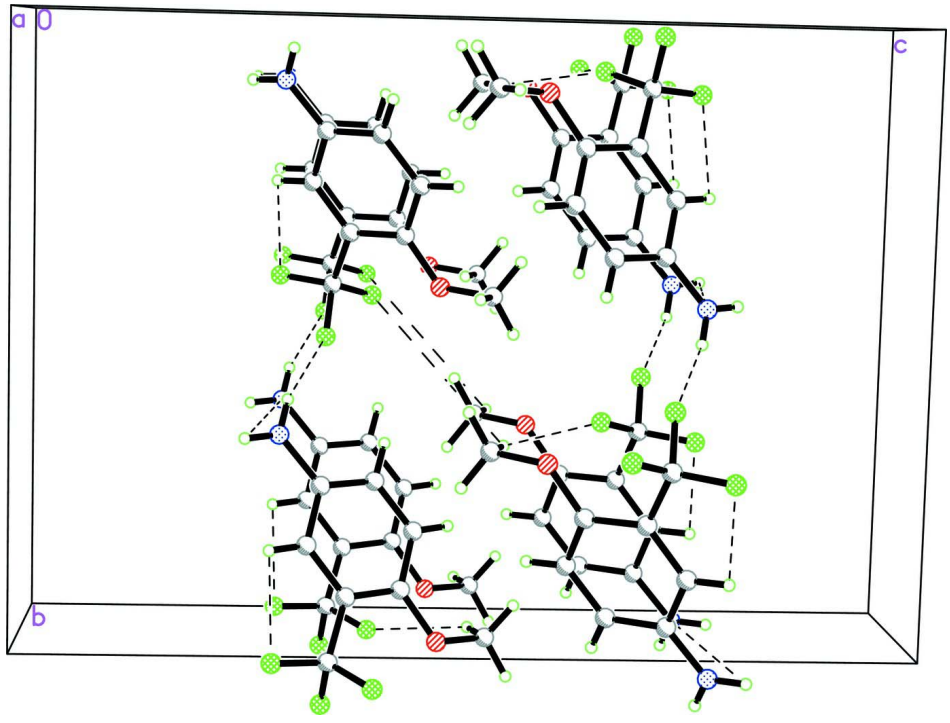

Figure 2

A packing diagram of the title compound showing hydrogen bonds dashed lines.

4-Methoxy-3-(trifluoromethyl)aniline

Crystal data

$C_8H_8F_3NO$
$M_r = 191.15$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 5.4140$ (11) Å
$b = 14.880$ (3) Å
$c = 21.304$ (4) Å
$V = 1716.3$ (6) Å^3
$Z = 8$
$F(000) = 784$
$D_x = 1.480$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 3.3$–$20.0°$
$\mu = 0.14$ mm^{-1}
$T = 293$ K
Prism, colorless
0.10 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 28.5714 pixels mm^{-1}
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.986$, $T_{max} = 0.986$
1722 measured reflections
1722 independent reflections
1389 reflections with $I > 2\sigma(I)$
$R_{int} = 0.000$
$\theta_{max} = 26.4°$, $\theta_{min} = 3.3°$
$h = 0 \rightarrow 6$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 26$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.162$
$S = 1.13$
1722 reflections
118 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
$w = 1/[\sigma^2(F_o^2) + (0.074P)^2 + 0.3348P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}

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}
N	0.0910 (3)	0.09833 (11)	0.20799 (9)	0.0642 (5)
H0A	0.1826	0.0524	0.2003	0.077*
H0B	0.0020	0.0998	0.2414	0.077*
F3	−0.3126 (4)	0.39455 (11)	0.21116 (7)	0.1021 (6)

F2	−0.3656 (3)	0.42452 (10)	0.11387 (8)	0.0906 (5)
C7	−0.0486 (4)	0.32133 (13)	0.13969 (9)	0.0548 (5)
F1	−0.0537 (3)	0.47609 (9)	0.16277 (8)	0.0924 (6)
O	0.0805 (4)	0.39518 (12)	0.04733 (8)	0.0872 (6)
C6	−0.0515 (4)	0.24725 (13)	0.17919 (10)	0.0550 (5)
H6A	−0.1479	0.2487	0.2153	0.066*
C5	0.0868 (4)	0.17085 (13)	0.16592 (9)	0.0557 (5)
C8	−0.1937 (4)	0.40274 (15)	0.15659 (10)	0.0643 (6)
C2	0.0907 (4)	0.32022 (15)	0.08453 (10)	0.0653 (6)
C4	0.2268 (5)	0.17158 (16)	0.11146 (11)	0.0719 (7)
H4A	0.3216	0.1214	0.1016	0.086*
C3	0.2297 (5)	0.24452 (18)	0.07140 (11)	0.0767 (7)
H3A	0.3260	0.2427	0.0353	0.092*
C1	0.2463 (6)	0.4017 (2)	−0.00398 (11)	0.0865 (8)
H1A	0.2177	0.4570	−0.0260	0.130*
H1B	0.2203	0.3520	−0.0319	0.130*
H1C	0.4131	0.4005	0.0113	0.130*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N	0.0752 (12)	0.0474 (10)	0.0699 (11)	0.0025 (8)	0.0031 (9)	0.0081 (8)
F3	0.1383 (15)	0.0767 (10)	0.0912 (11)	0.0339 (10)	0.0413 (10)	0.0151 (8)
F2	0.0887 (10)	0.0811 (11)	0.1018 (11)	0.0177 (8)	−0.0227 (9)	0.0065 (8)
C7	0.0605 (11)	0.0477 (11)	0.0562 (11)	−0.0037 (9)	−0.0035 (10)	0.0018 (8)
F1	0.1106 (12)	0.0541 (8)	0.1124 (12)	−0.0104 (8)	−0.0073 (9)	−0.0123 (7)
O	0.1094 (14)	0.0758 (11)	0.0763 (11)	0.0142 (10)	0.0150 (10)	0.0304 (9)
C6	0.0620 (12)	0.0492 (11)	0.0539 (11)	−0.0033 (9)	0.0009 (10)	0.0014 (8)
C5	0.0625 (12)	0.0482 (11)	0.0563 (11)	−0.0046 (9)	−0.0058 (10)	0.0021 (8)
C8	0.0757 (15)	0.0539 (12)	0.0632 (12)	0.0042 (10)	0.0003 (11)	0.0069 (10)
C2	0.0782 (14)	0.0604 (13)	0.0572 (12)	0.0005 (11)	0.0006 (11)	0.0119 (10)
C4	0.0852 (16)	0.0629 (14)	0.0674 (13)	0.0141 (12)	0.0126 (13)	0.0032 (10)
C3	0.0922 (17)	0.0765 (16)	0.0614 (13)	0.0136 (13)	0.0179 (13)	0.0116 (11)
C1	0.0955 (18)	0.0942 (19)	0.0697 (15)	−0.0139 (15)	0.0077 (15)	0.0258 (14)

Geometric parameters (Å, º)

N—C5	1.403 (3)	C6—C5	1.390 (3)
N—H0A	0.8600	C6—H6A	0.9300
N—H0B	0.8600	C5—C4	1.386 (3)
F3—C8	1.335 (3)	C2—C3	1.383 (3)
F2—C8	1.342 (3)	C4—C3	1.381 (3)
C7—C6	1.387 (3)	C4—H4A	0.9300
C7—C2	1.396 (3)	C3—H3A	0.9300
C7—C8	1.488 (3)	C1—H1A	0.9600
F1—C8	1.335 (3)	C1—H1B	0.9600
O—C2	1.369 (3)	C1—H1C	0.9600
O—C1	1.418 (3)		

C5—N—H0A	120.0	F1—C8—C7	112.9 (2)
C5—N—H0B	120.0	F2—C8—C7	113.50 (19)
H0A—N—H0B	120.0	O—C2—C3	124.6 (2)
C6—C7—C2	120.48 (19)	O—C2—C7	117.1 (2)
C6—C7—C8	119.62 (19)	C3—C2—C7	118.27 (19)
C2—C7—C8	119.89 (18)	C3—C4—C5	122.0 (2)
C2—O—C1	118.4 (2)	C3—C4—H4A	119.0
C7—C6—C5	121.37 (19)	C5—C4—H4A	119.0
C7—C6—H6A	119.3	C4—C3—C2	120.6 (2)
C5—C6—H6A	119.3	C4—C3—H3A	119.7
C4—C5—C6	117.27 (18)	C2—C3—H3A	119.7
C4—C5—N	122.13 (19)	O—C1—H1A	109.5
C6—C5—N	120.53 (19)	O—C1—H1B	109.5
F3—C8—F1	105.23 (19)	H1A—C1—H1B	109.5
F3—C8—F2	106.14 (19)	O—C1—H1C	109.5
F1—C8—F2	105.25 (18)	H1A—C1—H1C	109.5
F3—C8—C7	113.03 (18)	H1B—C1—H1C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*C*···F2^{i}	0.96	2.52	3.292 (3)	138
C6—H6*A*···F3	0.93	2.35	2.696 (3)	102
N—H0*A*···F1^{ii}	0.86	2.44	3.242 (2)	155
N—H0*B*···N^{iii}	0.86	2.47	3.245 (3)	150

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1/2, y-1/2, z$; (iii) $x-1/2, y, -z+1/2$.