

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2,4,6-Trifluoroaniline

Richard Betz and Peter Klüfers*

Ludwig-Maximilians Universität, Department Chemie und Biochemie, Butenandtstrasse 5–13 (Haus D), 81377 München, Germany

Correspondence e-mail: kluef@cup.uni-muenchen.de

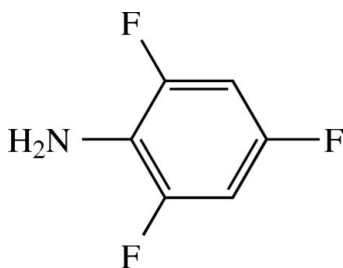
Received 10 September 2008; accepted 28 October 2008

Key indicators: single-crystal X-ray study; $T = 200$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.034; wR factor = 0.082; data-to-parameter ratio = 8.5.

The title compound, $\text{C}_6\text{H}_4\text{F}_3\text{N}$, is a fluoro derivative of aniline. The molecule shows non-crystallographic mirror symmetry. Bond lengths are normal. The $\text{C}-\text{C}-\text{C}$ angles show some deviation from the expected ideal values by up to 5° , a finding which is in accordance with a similar structure in the literature. In the crystal structure $\text{H}\cdots\text{F}$ contacts and $\text{H}\cdots\text{N}$ contacts lead to the formation of sheets whose surfaces are made up by the hydrophobic phenyl rings.

Related literature

For the crystal structure of a related compound, see: Gdaniec (2007). For graph-set analysis, see: Bernstein *et al.* (1995); Etter *et al.* (1990).



Experimental

Crystal data

 $\text{C}_6\text{H}_4\text{F}_3\text{N}$
 $M_r = 147.10$

 Orthorhombic, $P2_12_12_1$
 $a = 6.3220$ (6) Å

 $b = 24.792$ (2) Å
 $c = 3.8545$ (5) Å
 $V = 604.14$ (11) Å³
 $Z = 4$

 Mo $K\alpha$ radiation
 $\mu = 0.16$ mm⁻¹
 $T = 200$ (2) K
 $0.50 \times 0.09 \times 0.05$ mm

Data collection

 Oxford Diffraction KappaCCD diffractometer
 Absorption correction: multi-scan (SCALE3 ABSPACK in *CrysAlis RED*; Oxford Diffraction, 2005)
 $T_{\min} = 0.921$, $T_{\max} = 0.992$

 4517 measured reflections
 775 independent reflections
 489 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.082$
 $S = 0.94$
 775 reflections

 91 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.13$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H71}\cdots\text{N1}^i$	0.90	2.26	3.110 (3)	157
$\text{N1}-\text{H72}\cdots\text{F3}^{ii}$	0.88	2.31	3.086 (2)	147

Symmetry codes: (i) $-x + \frac{3}{2}, -y, z - \frac{1}{2}$; (ii) $-x + \frac{1}{2}, -y, z - \frac{1}{2}$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2005); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2005); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

The authors thank Dr Peter Mayer for professional support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RN2051).

References

- Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
- Burnett, M. N. & Johnson, C. K. (1996). *ORTEPIII*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
- Etter, M. C., MacDonald, J. C. & Bernstein, J. (1990). *Acta Cryst.* **B46**, 256–262.
- Gdaniec, M. (2007). *Acta Cryst.* **E63**, o2954.
- Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
- Oxford Diffraction (2005). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, England.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supporting information

Acta Cryst. (2008). E64, o2242 [doi:10.1107/S1600536808035083]

2,4,6-Trifluoroaniline

Richard Betz and Peter Klüfers

S1. Comment

In a program focused on the synthesis of derivatives of phenylarsonic acid a number of substituted aniline-derivatives were chosen as starting materials. In order to compare the influence of an arsonic group on the geometry of these starting materials, the crystal structure of 2,4,6-trifluoroaniline was elucidated by means of single-crystal X-ray diffraction.

In the molecule (Fig. 1) the C–C–C angles deviate from the expected ideal value of 120° by up to 5°. Angles bigger than the expected value are invariably found at C atoms bonded to fluorine, the smallest angle being present on the C atom bearing the amino group. This finding is in agreement with the situation observed in 2,3,4,5,6-pentafluoroaniline (Gdaniec, 2007).

In the crystal structure hydrogen bonds between fluorine and the amino group are present. If contacts whose ranges fall 0.2 Å below the sum of van der Waals radii of the respective atoms are taken into consideration, only one of the F atoms in *ortho* position to the amino group is participating in these intermolecular interactions. These connect the molecules into sheets parallel to [1 0 1]. The surfaces of these sheets are made up by the aromatic moieties (Fig. 2 and Fig. 3). In terms of graph-set analysis (Etter *et al.*, 1990; Bernstein *et al.*, 1995) the N–H···F pattern should be assigned a C(5) descriptor on the unitary level while the remaining H atom on nitrogen participates in a cooperative chain of hydrogen bonds (N–H···N).

S2. Experimental

The compound was obtained commercially from Fluorochem. Crystals suitable for X-ray diffraction studies were obtained upon cooling the compound to 4 °C in a fridge.

S3. Refinement

All H atoms were located in a difference map and refined as riding on their parent atoms with $U_{\text{iso}}(\text{H})$ values of 1.2 $U_{\text{eq}}(\text{C})$ and 1.2 $U_{\text{eq}}(\text{N})$.

Due to the absence of a significant anomalous scatterer in the molecule, the Flack parameter is meaningless. Friedel opposites were merged and the absolute structure parameter was removed from the CIF file.

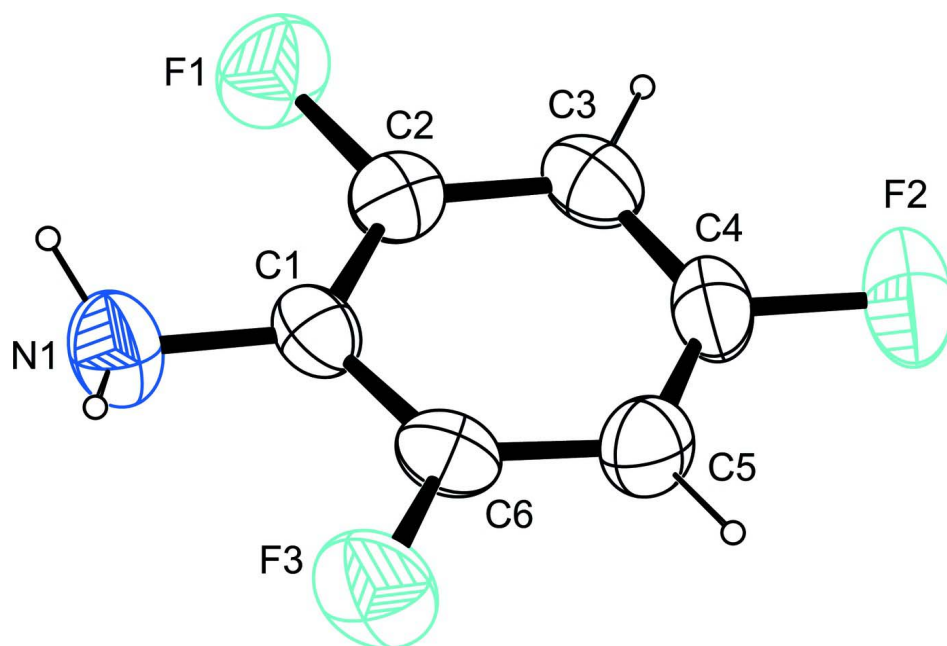


Figure 1

The molecular structure of the title compound, with atom labels and anisotropic displacement ellipsoids (drawn at 50% probability level) for non-H atoms.

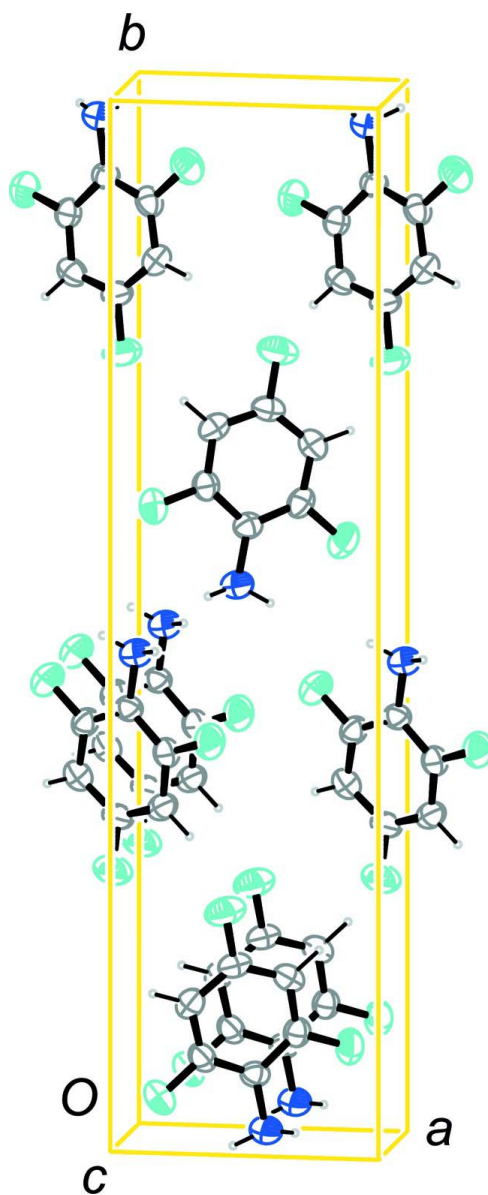
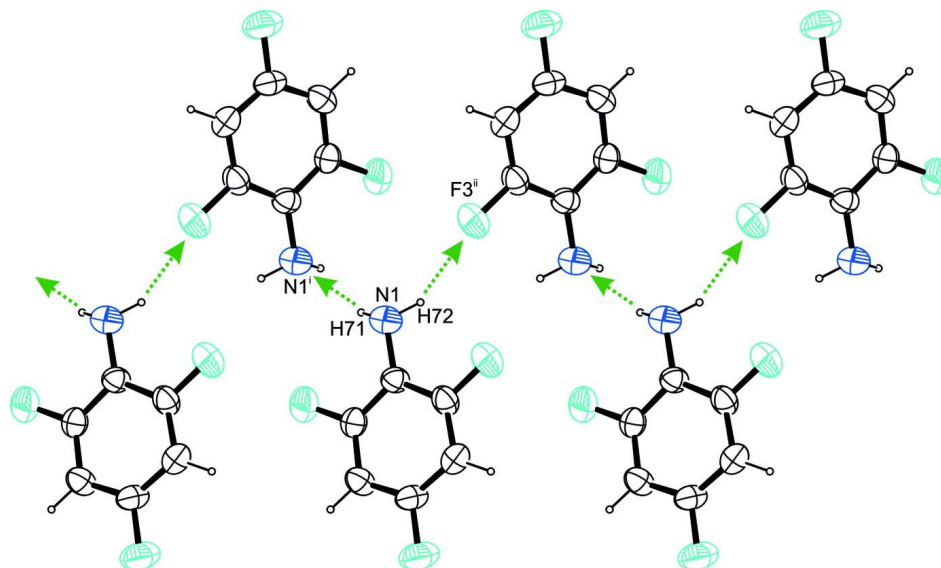


Figure 2

The packing of the title compound, viewed along $[0\ 0\ -1]$.

**Figure 3**

Intermolecular interactions in the crystal structure of the title compound, viewed along [0 0 1]. Symmetry operators: i $-x + 3/2, -y, z - 1/2$; ii $-x + 1/2, -y, z - 1/2$.

2,4,6-Trifluoroaniline

Crystal data

$C_6H_4F_3N$

$M_r = 147.10$

Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

$a = 6.3220$ (6) Å

$b = 24.792$ (2) Å

$c = 3.8545$ (5) Å

$V = 604.14$ (11) Å³

$Z = 4$

$F(000) = 296$

$D_x = 1.617$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1719 reflections

$\theta = 4.1$ – 26.3°

$\mu = 0.16$ mm⁻¹

$T = 200$ K

Rod, colourless

$0.50 \times 0.09 \times 0.05$ mm

Data collection

Oxford Diffraction KappaCCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan

(SCALE3 ABSPACK in *CrysAlis RED*; Oxford
Diffraction, 2005)

$T_{\min} = 0.921$, $T_{\max} = 0.992$

4517 measured reflections

775 independent reflections

489 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

$\theta_{\max} = 26.4^\circ$, $\theta_{\min} = 4.1^\circ$

$h = -7 \rightarrow 7$

$k = -30 \rightarrow 30$

$l = -4 \rightarrow 3$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.034$

$wR(F^2) = 0.082$

$S = 0.94$

775 reflections

91 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: difference Fourier map
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0505P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.13 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.16 \text{ e } \text{Å}^{-3}$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
F1	0.8818 (2)	0.10411 (5)	-0.1028 (5)	0.0618 (5)
F2	0.4694 (2)	0.24248 (5)	0.3783 (5)	0.0712 (6)
F3	0.2254 (2)	0.06360 (6)	0.3828 (5)	0.0715 (6)
N1	0.5886 (3)	0.02701 (7)	0.0726 (7)	0.0564 (7)
H71	0.6769	0.0202	-0.1054	0.068*
H72	0.4647	0.0109	0.0632	0.068*
C1	0.5551 (4)	0.08172 (8)	0.1384 (7)	0.0398 (6)
C2	0.7034 (4)	0.12061 (9)	0.0624 (7)	0.0414 (6)
C3	0.6805 (4)	0.17436 (8)	0.1376 (7)	0.0447 (7)
H3	0.7868	0.2000	0.0811	0.054*
C4	0.4963 (4)	0.18920 (8)	0.2983 (7)	0.0451 (7)
C5	0.3394 (4)	0.15382 (8)	0.3828 (8)	0.0480 (7)
H5	0.2124	0.1652	0.4922	0.058*
C6	0.3757 (4)	0.10062 (9)	0.3004 (7)	0.0444 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0434 (9)	0.0688 (9)	0.0731 (11)	0.0014 (7)	0.0104 (9)	-0.0040 (10)
F2	0.0870 (11)	0.0420 (8)	0.0845 (14)	0.0118 (8)	-0.0120 (14)	-0.0085 (10)
F3	0.0534 (9)	0.0641 (9)	0.0969 (14)	-0.0156 (7)	0.0105 (12)	0.0123 (10)
N1	0.0574 (14)	0.0408 (11)	0.0712 (18)	-0.0029 (10)	-0.0014 (15)	-0.0017 (12)
C1	0.0441 (13)	0.0354 (12)	0.0397 (16)	-0.0004 (10)	-0.0070 (14)	0.0045 (12)
C2	0.0379 (13)	0.0476 (14)	0.0386 (16)	0.0022 (11)	-0.0023 (13)	0.0011 (13)
C3	0.0503 (17)	0.0412 (14)	0.0426 (17)	-0.0103 (11)	-0.0064 (16)	0.0057 (14)
C4	0.0573 (16)	0.0326 (12)	0.0453 (17)	0.0063 (12)	-0.0101 (16)	-0.0018 (12)
C5	0.0445 (16)	0.0496 (14)	0.0499 (17)	0.0094 (12)	0.0004 (16)	0.0009 (16)
C6	0.0408 (13)	0.0473 (14)	0.0453 (19)	-0.0087 (12)	0.0001 (14)	0.0094 (13)

Geometric parameters (Å , $^\circ$)

F1—C2	1.358 (3)	C1—C6	1.377 (3)
F2—C4	1.367 (2)	C2—C3	1.371 (3)
F3—C6	1.359 (2)	C3—C4	1.369 (4)
N1—C1	1.396 (3)	C3—H3	0.9500
N1—H71	0.9009	C4—C5	1.364 (3)
N1—H72	0.8798	C5—C6	1.376 (3)
C1—C2	1.377 (3)	C5—H5	0.9500
C1—N1—H71	114.6	C2—C3—H3	121.7
C1—N1—H72	108.3	C5—C4—F2	118.5 (2)

H71—N1—H72	115.8	C5—C4—C3	123.6 (2)
C2—C1—C6	114.8 (2)	F2—C4—C3	117.9 (2)
C2—C1—N1	122.6 (2)	C4—C5—C6	116.1 (2)
C6—C1—N1	122.5 (2)	C4—C5—H5	121.9
F1—C2—C3	118.7 (2)	C6—C5—H5	121.9
F1—C2—C1	117.04 (19)	F3—C6—C5	118.5 (2)
C3—C2—C1	124.3 (2)	F3—C6—C1	116.9 (2)
C4—C3—C2	116.5 (2)	C5—C6—C1	124.6 (2)
C4—C3—H3	121.7		
C6—C1—C2—F1	-178.9 (2)	F2—C4—C5—C6	-179.2 (2)
N1—C1—C2—F1	4.2 (4)	C3—C4—C5—C6	0.7 (4)
C6—C1—C2—C3	0.0 (4)	C4—C5—C6—F3	179.2 (3)
N1—C1—C2—C3	-176.9 (3)	C4—C5—C6—C1	-0.8 (4)
F1—C2—C3—C4	178.8 (2)	C2—C1—C6—F3	-179.5 (2)
C1—C2—C3—C4	-0.1 (4)	N1—C1—C6—F3	-2.7 (4)
C2—C3—C4—C5	-0.2 (4)	C2—C1—C6—C5	0.5 (4)
C2—C3—C4—F2	179.6 (2)	N1—C1—C6—C5	177.3 (3)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H71 \cdots N1 ⁱ	0.90	2.26	3.110 (3)	157
N1—H72 \cdots F3 ⁱⁱ	0.88	2.31	3.086 (2)	147

Symmetry codes: (i) $-x+3/2, -y, z-1/2$; (ii) $-x+1/2, -y, z-1/2$.