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4-Aminophenylsulfur pentafluoride

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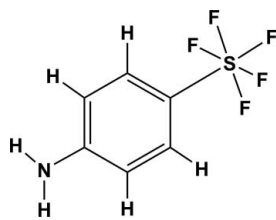
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Key indicators: single-crystal X-ray study; $T = 200$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å;
R factor = 0.031; wR factor = 0.066; data-to-parameter ratio = 14.0.

In the title compound, $\text{C}_6\text{H}_6\text{F}_5\text{NS}$, the environment of the S atom is roughly octahedral. The axial F—S bond appears slightly elongated with respect to the four equatorial F—S bonds. Equatorial F atoms are staggered with respect to the benzene ring. The N atom is displaced from the benzene plane by 0.154 (4) Å. The F—S—C—C torsion angles differ greatly from the values observed in the related structure of 4-acetamidophenylsulfur pentafluoride. The packing is stabilized by weak N—H...F contacts.

Related literature

For related literature, see: Raasch (1963); Bowden *et al.* (2000); Sheppard (1960, 1962).



Experimental

Crystal data

$\text{C}_6\text{H}_6\text{F}_5\text{NS}$
 $M_r = 219.18$
Orthorhombic, *Pbca*
 $a = 16.0369$ (13) Å
 $b = 5.7514$ (5) Å
 $c = 17.5305$ (15) Å

$V = 1616.9$ (2) Å³
 $Z = 8$
Mo $K\alpha$ radiation
 $\mu = 0.44$ mm⁻¹
 $T = 200$ K
 $0.1 \times 0.08 \times 0.05$ mm

Data collection

Rigaku Mercury CCD
diffractometer
Absorption correction: multi-scan
(Blessing, 1995)
 $T_{\min} = 0.959$, $T_{\max} = 0.981$

6533 measured reflections
1650 independent reflections
633 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.051$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.065$
 $S = 0.58$
1650 reflections

118 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.26$ e Å⁻³
 $\Delta\rho_{\min} = -0.22$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H12...F5 ⁱ	0.89	2.59	3.38	148

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

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-III* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *enCIFer* (Allen *et al.*, 2004).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2303).

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supporting information

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4-Aminophenylsulfur pentafluoride

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S1. Comment

Phenylsulfur pentafluorides were first synthesized (Sheppard, 1960) by the fluorination of aromatic disulfides with silver difluoride. Some SF₅-benzene derivatives were patented as plant regulants, herbicides and bactericides (Raasch, 1963).

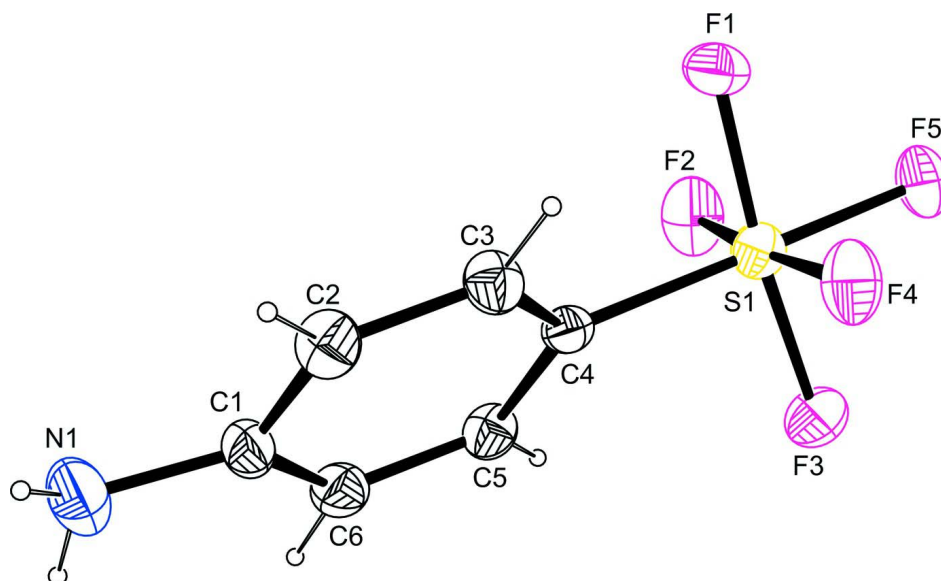
In the title compound, the environment of sulfur atom appears to be approximately octahedral (Fig. 1) with the C – S bond being 1.786 (3) Å, four equatorial S – F bonds of 1.577 (2) – 1.586 (2) Å and noticeably elongated to 1.600 (2) Å axial S – F bond. Equatorial F atoms are declined slightly away from the benzene ring resulting in the medium value of Feq – S – Fax angle of 86.9 °. Similar staggered conformation was observed earlier in the structure of 4-acetamidophenylsulfur pentafluoride (Bowden *et al.*, 2000). The F – S – C – C dihedral angles values of 43 and 47 ° differ from observed in above mentioned structure of 4-acetamidophenylsulfur pentafluoride 30 and 60 ° respectively. The packing is stabilized by weak N—H···F contacts.

S2. Experimental

Sample of 4-aminophenylsulfur pentafluoride was prepared in three steps according to original procedure (Sheppard, 1962). Bis-(4-nitrophenyl)-disulfide was fluorinated with silver difluoride in CFC113 solvent and the product 4-nitrophenylsulfur-pentafluoride was obtained in 10.0% yield and was consequently purified by preparative HPLC. 95% pure 4-nitrophenylsulfur pentafluoride was hydrogenated with hydrogen gas in acidic (HCL) ethanol solution, PtO₂ was used as a catalyst. The 4-aminophenylsulfur pentafluoride hydrochloride obtained was reacted with sodium bicarbonate water solution and the product 4-aminophenylsulfur pentafluoride was extracted with diethyl ether and recrystallized from pentane. 4-Aminophenylsulfur pentafluoride crystallizes as white needles.

S3. Refinement

All H atoms attached to C atoms were fixed geometrically and treated as riding with C—H = 0.93 Å (aromatic) with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. H atoms of amino group were located in difference Fourier maps and included in the subsequent refinement using restraints (N—H = 0.89 (1) Å and H···H = 1.57 (2) Å) with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. In the last stage of refinement, they were treated as riding on their parent N atom.

**Figure 1**

Molecular view of I with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

4-Aminophenylsulfur pentafluoride

Crystal data

$C_6H_6F_5NS$

$M_r = 219.18$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 16.0369$ (13) Å

$b = 5.7514$ (5) Å

$c = 17.5305$ (15) Å

$V = 1616.9$ (2) Å³

$Z = 8$

$F(000) = 880$

$D_x = 1.801$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å

Cell parameters from 71 reflections

$\theta = 1.2$ – 29.1°

$\mu = 0.44$ mm⁻¹

$T = 200$ K

Chunk, colourless

$0.1 \times 0.08 \times 0.05$ mm

Data collection

Mercury CCD (2x2 bin mode)
diffractometer

dtprofit.ref scans

Absorption correction: multi-scan
(Blessing, 1995)

$T_{\min} = 0.959$, $T_{\max} = 0.981$

6533 measured reflections

1650 independent reflections

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

$R_{\text{int}} = 0.051$

$\theta_{\max} = 26.4^\circ$, $\theta_{\min} = 2.3^\circ$

$h = 0 \rightarrow 20$

$k = 0 \rightarrow 7$

$l = 0 \rightarrow 21$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.031$

$wR(F^2) = 0.065$

$S = 0.58$

1650 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

$w = 1/[\sigma^2(F_o^2)]$

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

$$\Delta\rho_{\max} = 0.26 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.22 \text{ e } \text{\AA}^{-3}$$

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.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.89753 (5)	0.08028 (15)	0.09772 (6)	0.0401 (2)
F1	0.87251 (10)	0.3449 (3)	0.10747 (12)	0.0606 (6)
F2	0.80397 (8)	0.0204 (3)	0.07597 (11)	0.0597 (6)
F3	0.92307 (9)	-0.1787 (3)	0.07811 (11)	0.0529 (6)
F4	0.99217 (9)	0.1448 (3)	0.10997 (12)	0.0566 (6)
F5	0.91119 (10)	0.1370 (3)	0.00922 (11)	0.0608 (6)
C1	0.84866 (18)	-0.0742 (6)	0.3495 (2)	0.0417 (9)
C2	0.89380 (16)	0.1215 (6)	0.3276 (2)	0.0424 (9)
H2	0.9136	0.2228	0.3648	0.051*
C3	0.90968 (17)	0.1681 (5)	0.2514 (2)	0.0391 (9)
H3	0.9394	0.3005	0.2377	0.047*
C4	0.88150 (16)	0.0181 (5)	0.19639 (18)	0.0303 (8)
C5	0.83895 (16)	-0.1814 (5)	0.2167 (2)	0.0364 (8)
H5	0.8205	-0.2842	0.1794	0.044*
C6	0.82413 (17)	-0.2267 (5)	0.2925 (2)	0.0415 (9)
H6	0.7969	-0.3635	0.3059	0.050*
N1	0.82569 (15)	-0.1078 (5)	0.42463 (17)	0.0618 (9)
H11	0.8081	-0.2512	0.4320	0.074*
H12	0.8522	-0.0289	0.4603	0.074*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0450 (5)	0.0386 (5)	0.0367 (6)	0.0025 (4)	0.0026 (5)	-0.0008 (5)
F1	0.0980 (14)	0.0353 (11)	0.0486 (15)	0.0151 (10)	0.0129 (12)	0.0070 (11)
F2	0.0412 (10)	0.0926 (15)	0.0453 (15)	-0.0039 (9)	-0.0090 (10)	-0.0005 (12)
F3	0.0731 (12)	0.0369 (11)	0.0487 (15)	0.0077 (9)	0.0085 (11)	-0.0127 (10)
F4	0.0432 (10)	0.0714 (13)	0.0552 (16)	-0.0147 (9)	0.0108 (10)	-0.0010 (12)
F5	0.0819 (13)	0.0706 (14)	0.0300 (13)	0.0071 (10)	0.0147 (11)	0.0067 (11)
C1	0.040 (2)	0.053 (2)	0.032 (2)	0.0120 (17)	0.0036 (18)	0.009 (2)
C2	0.0417 (19)	0.047 (2)	0.038 (2)	-0.0027 (17)	-0.0073 (18)	-0.0070 (19)
C3	0.0419 (19)	0.037 (2)	0.038 (2)	-0.0076 (15)	0.0024 (18)	-0.0009 (19)
C4	0.0336 (17)	0.0276 (18)	0.030 (2)	0.0041 (14)	0.0002 (15)	-0.0005 (16)
C5	0.0362 (18)	0.0312 (19)	0.042 (2)	-0.0041 (15)	0.0018 (17)	-0.0071 (19)
C6	0.0424 (19)	0.033 (2)	0.049 (3)	-0.0003 (16)	0.0081 (19)	0.004 (2)
N1	0.0770 (19)	0.068 (2)	0.040 (2)	-0.0009 (16)	0.0052 (17)	0.0081 (19)

Geometric parameters (Å, °)

S1—F4	1.5771 (16)	C2—H2	0.9300
S1—F3	1.5826 (17)	C3—C4	1.370 (4)
S1—F1	1.5832 (17)	C3—H3	0.9300
S1—F2	1.5860 (16)	C4—C5	1.382 (4)
S1—F5	1.600 (2)	C5—C6	1.375 (4)
S1—C4	1.785 (3)	C5—H5	0.9300
C1—N1	1.381 (4)	C6—H6	0.9300
C1—C6	1.386 (4)	N1—H11	0.8813
C1—C2	1.392 (4)	N1—H12	0.8823
C2—C3	1.386 (4)		
F4—S1—F3	90.11 (9)	C3—C2—C1	121.2 (3)
F4—S1—F1	90.18 (10)	C3—C2—H2	119.4
F3—S1—F1	173.62 (13)	C1—C2—H2	119.4
F4—S1—F2	173.86 (13)	C4—C3—C2	119.7 (3)
F3—S1—F2	89.33 (10)	C4—C3—H3	120.1
F1—S1—F2	89.70 (10)	C2—C3—H3	120.1
F4—S1—F5	87.26 (10)	C3—C4—C5	120.2 (3)
F3—S1—F5	86.90 (11)	C3—C4—S1	120.6 (2)
F1—S1—F5	86.75 (11)	C5—C4—S1	119.2 (3)
F2—S1—F5	86.60 (11)	C6—C5—C4	119.5 (3)
F4—S1—C4	93.09 (12)	C6—C5—H5	120.2
F3—S1—C4	93.40 (12)	C4—C5—H5	120.2
F1—S1—C4	92.95 (13)	C5—C6—C1	121.8 (3)
F2—S1—C4	93.05 (12)	C5—C6—H6	119.1
F5—S1—C4	179.54 (13)	C1—C6—H6	119.1
N1—C1—C6	121.5 (3)	C1—N1—H11	110.9
N1—C1—C2	121.0 (4)	C1—N1—H12	118.5
C6—C1—C2	117.4 (3)	H11—N1—H12	122.1

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H12...F5 ⁱ	0.89	2.59	3.38	148

Symmetry code: (i) *x*, $-y+1/2$, *z*+1/2.