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Bis[4-(2-nitrobenzenesulfonamido)-pyridinium] hexafluorosilicate

Ai-Rong Wang

School of Chemistry and Chemical Engineering, Henan Institute of Science and Technology, Xinxiang 453003, People's Republic of China
Correspondence e-mail: airong_wang@yahoo.cn

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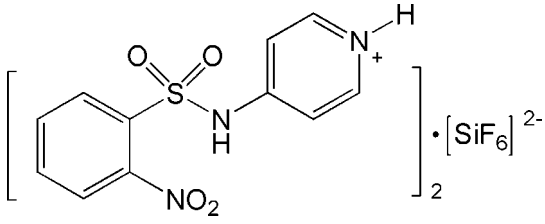
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.043; wR factor = 0.111; data-to-parameter ratio = 14.5.

In the title compound, $2C_{11}H_{10}N_3O_4S^+\cdot SiF_6^{2-}$, the short C—N distance [1.386 (2) Å] is indicative of a slight conjugation of N with the π electrons of the pyridinium ring, and with those of the sulfonyl group. The dihedral angle between the benzene and pyridinium rings is 77.48 (7)°. The crystal structure is stabilized by N—H···F and C—H···F hydrogen bonds. The Si atom of the anion lies on a special position.

Related literature

For zwitterionic forms of *N*-arylbenzenesulfonamides, see: Li *et al.* (2007); Yu & Li (2007). For reference geometric data, see: Allen *et al.* (1987). Damiano *et al.* (2007) describe the use of pyridinium derivatives for the construction of supramolecular architectures.



Experimental

Crystal data

$2C_{11}H_{10}N_3O_4S^+\cdot SiF_6^{2-}$ M_r = 702.65
Monoclinic, $C2/c$
a = 22.691 (5) Å
b = 8.524 (2) Å
c = 14.776 (3) Å
β = 110.95 (3)°
V = 2669 (1) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.35 mm^{-1}
T = 113 (2) K
0.22 × 0.16 × 0.04 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.927, T_{max} = 0.986
10620 measured reflections
3064 independent reflections
2456 reflections with $I > 2\sigma(I)$
R_{int} = 0.053

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.111
S = 1.06
3064 reflections
212 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···F1^{i}	0.86 (3)	1.96 (3)	2.789 (2)	162 (3)
N2—H2N···F1^{ii}	0.73 (3)	1.97 (3)	2.690 (2)	171 (3)
C4—H4···F2^{i}	0.95	2.35	3.141 (2)	141
C5—H5···F3^{ii}	0.95	2.50	3.426 (3)	165

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2077).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Damiano, T., Morton, D. & Nelson, A. (2007). *Org. Biomol. Chem.* **5**, 2735—2752.
Li, J. S., Chen, L. G., Zhang, Y. Y., Xu, Y. J., Deng, Y. & Huang, P. M. (2007). *J. Chem. Res.* **6**, 350–352.
Rigaku/MSC (2005). *CrystalClear*. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Yu, H.-J. & Li, J.-S. (2007). *Acta Cryst.* E**63**, o3399.

supporting information

Acta Cryst. (2008). E**64**, o2405 [doi:10.1107/S1600536808038191]

Bis[4-(2-nitrobenzenesulfonamido)pyridinium] hexafluorosilicate

Ai-Rong Wang

S1. Comment

Organic pyridinium salts have been widely used in the construction of supramolecular architectures (Damiano *et al.*, 2007). As part of our ongoing studies of supramolecular chemistry involving the pyridinium rings (Li *et al.*, 2007), herein we report the crystal structure of the title compound, 4-(2-nitrophenylsulfonylamino)pyridinium hexafluorosilicate (Fig. 1).

In the cations of the title compound the short C—N distance [N2—C1 = 1.386 (2) Å] has a value between those of a typical C═N double and C—N single bond (1.47–1.50 Å and 1.34–1.38 Å, respectively; Allen *et al.*, 1987). This might be indicative of a slight conjugation of N with π electrons of the pyridinium ring, and with those of the sulfonyl group. The dihedral angle between the benzene ring and the pyridinium ring is 77.48 (7)°. The crystal structure is stabilized by N—H···F and C—H···F hydrogen bonds (Table 1).

S2. Experimental

A solution of 2-nitrobenzenesulfonyl chloride (2.2 g, 10 mmol) in CH_2Cl_2 (10 ml) was added dropwise to a suspension of 4-aminopyridine (0.9 g, 10 mmol) in CH_2Cl_2 (10 ml) at room temperature with stirring. The reaction mixture was stirred overnight. The yellow solid obtained was washed with warm water to obtain the title compound in a yield of 55.3%. A colorless single crystal suitable for X-ray analysis was obtained by slow evaporation of a fluorosilicic acid (10%) solution at room temperature over a period of a week.

S3. Refinement

The N-bound H atoms were located in a difference density Fourier map and freely refined. The C-bound H atoms were positioned geometrically (C—H = 0.95 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C).

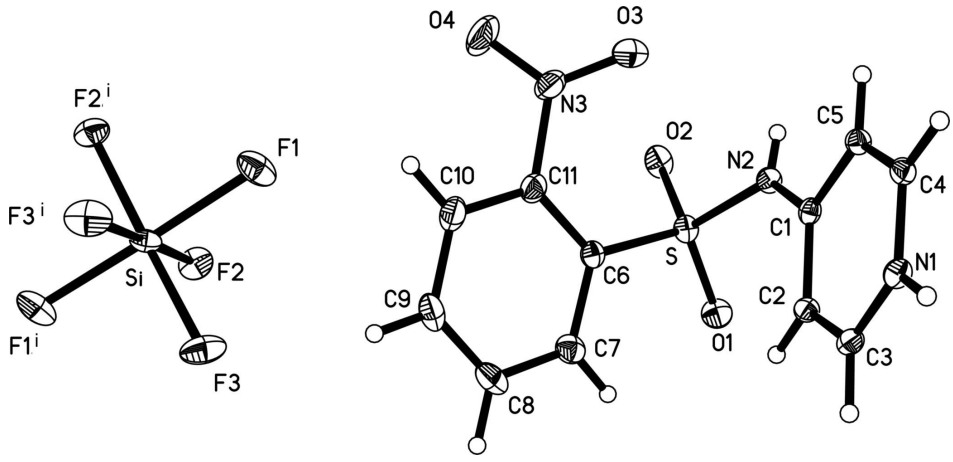


Figure 1

View of one molecule of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 35% probability level (arbitrary spheres for the H atoms). [Symmetry code: (i) -*x* + 2, *y*, -*z* + 3/2.]

Bis[4-(2-nitrobenzenesulfonamido)pyridinium] hexafluorosilicate

Crystal data

$2C_{11}H_{10}N_3O_4S^+\cdot SiF_6^{2-}$
M_r = 702.65
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 22.691 (5) Å
b = 8.524 (2) Å
c = 14.776 (3) Å
β = 110.95 (3)°
V = 2669 (1) Å^3
Z = 4
F(000) = 1432
D_x = 1.749 Mg m^{-3}
Mo *K*α radiation, λ = 0.71073 Å
Cell parameters from 3772 reflections
θ = 1.9–27.5°
μ = 0.35 mm^{-1}
T = 113 K
Block, colourless
0.22 × 0.16 × 0.04 mm

Data collection

Rigaku Saturn CCD area-detector diffractometer
Radiation source: rotating anode
Confocal monochromator
Detector resolution: 7.31 pixels mm^{-1}
ω and φ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.927, T_{max} = 0.986
10620 measured reflections
3064 independent reflections
2456 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
θ_{max} = 27.5°, θ_{min} = 1.9°
h = −22→29
k = −10→11
l = −18→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.111
S = 1.06
3064 reflections
212 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0513P)^2 + 1.0092P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.49 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S	0.61983 (2)	0.29097 (5)	0.52758 (4)	0.01969 (15)
O1	0.58452 (8)	0.32136 (18)	0.42794 (11)	0.0273 (4)
O2	0.63133 (8)	0.13308 (16)	0.56235 (12)	0.0282 (4)
O3	0.69576 (9)	0.3188 (2)	0.75056 (13)	0.0473 (5)
O4	0.78705 (10)	0.2230 (3)	0.7737 (2)	0.0843 (10)
N1	0.56160 (8)	0.8430 (2)	0.63088 (13)	0.0192 (4)
H1N	0.5583 (14)	0.942 (3)	0.639 (2)	0.052 (9)*
N2	0.58416 (9)	0.3725 (2)	0.59405 (14)	0.0194 (4)
H2N	0.5795 (14)	0.315 (3)	0.628 (2)	0.043 (9)*
N3	0.74149 (10)	0.3008 (2)	0.72703 (15)	0.0363 (5)
C1	0.57577 (9)	0.5317 (2)	0.60418 (14)	0.0167 (4)
C2	0.57532 (9)	0.6398 (2)	0.53308 (14)	0.0177 (4)
H2	0.5800	0.6058	0.4748	0.021*
C3	0.56798 (9)	0.7955 (2)	0.54852 (15)	0.0193 (4)
H3	0.5674	0.8704	0.5006	0.023*
C4	0.56074 (9)	0.7410 (2)	0.70051 (14)	0.0195 (4)
H4	0.5555	0.7784	0.7577	0.023*
C5	0.56742 (9)	0.5837 (2)	0.68832 (14)	0.0185 (4)
H5	0.5664	0.5109	0.7364	0.022*
C6	0.69299 (10)	0.3905 (2)	0.55358 (15)	0.0192 (4)
C7	0.70103 (11)	0.4743 (2)	0.47788 (16)	0.0258 (5)
H7	0.6675	0.4795	0.4169	0.031*
C8	0.75749 (12)	0.5503 (3)	0.49066 (18)	0.0309 (5)
H8	0.7620	0.6078	0.4385	0.037*
C9	0.80672 (11)	0.5435 (3)	0.57748 (19)	0.0307 (5)
H9	0.8451	0.5965	0.5854	0.037*
C10	0.80059 (10)	0.4591 (3)	0.65379 (18)	0.0295 (5)
H10	0.8347	0.4530	0.7141	0.035*
C11	0.74405 (10)	0.3839 (3)	0.64084 (16)	0.0249 (5)
Si	1.0000	0.65155 (9)	0.7500	0.01731 (19)
F1	0.94232 (7)	0.64561 (14)	0.79977 (10)	0.0303 (3)
F2	0.96119 (6)	0.51233 (14)	0.67115 (9)	0.0265 (3)
F3	0.96052 (7)	0.79039 (14)	0.67359 (9)	0.0333 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0207 (3)	0.0158 (3)	0.0229 (3)	−0.0010 (2)	0.0083 (2)	−0.00471 (19)
O1	0.0294 (9)	0.0304 (8)	0.0199 (8)	−0.0032 (7)	0.0060 (6)	−0.0071 (6)
O2	0.0324 (9)	0.0145 (7)	0.0426 (10)	0.0012 (7)	0.0196 (8)	−0.0020 (7)
O3	0.0360 (11)	0.0728 (14)	0.0333 (11)	0.0026 (10)	0.0127 (8)	0.0205 (10)
O4	0.0312 (11)	0.112 (2)	0.104 (2)	0.0222 (13)	0.0172 (12)	0.0855 (17)
N1	0.0217 (9)	0.0125 (8)	0.0228 (10)	0.0012 (7)	0.0073 (7)	−0.0012 (7)
N2	0.0221 (9)	0.0136 (8)	0.0242 (10)	0.0014 (7)	0.0104 (7)	0.0011 (7)
N3	0.0227 (10)	0.0443 (13)	0.0350 (12)	−0.0019 (9)	0.0019 (9)	0.0175 (10)
C1	0.0135 (9)	0.0149 (9)	0.0205 (10)	−0.0005 (8)	0.0045 (7)	−0.0011 (8)
C2	0.0195 (10)	0.0196 (10)	0.0138 (10)	0.0015 (8)	0.0060 (8)	−0.0017 (8)
C3	0.0184 (10)	0.0182 (10)	0.0195 (10)	0.0009 (8)	0.0048 (8)	0.0026 (8)
C4	0.0178 (10)	0.0244 (10)	0.0165 (10)	0.0018 (9)	0.0064 (8)	−0.0008 (8)
C5	0.0177 (10)	0.0195 (10)	0.0182 (10)	−0.0008 (8)	0.0063 (8)	0.0003 (8)
C6	0.0202 (10)	0.0168 (9)	0.0219 (11)	0.0020 (8)	0.0092 (8)	−0.0028 (8)
C7	0.0293 (12)	0.0270 (11)	0.0237 (12)	0.0011 (10)	0.0126 (9)	−0.0029 (9)
C8	0.0367 (14)	0.0297 (12)	0.0359 (14)	−0.0016 (11)	0.0247 (11)	−0.0002 (10)
C9	0.0225 (11)	0.0292 (11)	0.0457 (15)	−0.0018 (10)	0.0184 (10)	−0.0011 (11)
C10	0.0187 (11)	0.0289 (11)	0.0377 (14)	0.0019 (10)	0.0060 (9)	0.0040 (10)
C11	0.0216 (11)	0.0230 (10)	0.0295 (12)	0.0044 (9)	0.0083 (9)	0.0044 (9)
Si	0.0263 (4)	0.0132 (4)	0.0144 (4)	0.000	0.0098 (3)	0.000
F1	0.0431 (8)	0.0202 (6)	0.0393 (8)	0.0036 (6)	0.0292 (7)	0.0010 (5)
F2	0.0306 (7)	0.0222 (6)	0.0233 (7)	−0.0018 (6)	0.0056 (5)	−0.0054 (5)
F3	0.0505 (9)	0.0225 (7)	0.0248 (7)	0.0093 (6)	0.0110 (6)	0.0072 (5)

Geometric parameters (Å, º)

S—O1	1.426 (2)	C4—H4	0.9500
S—O2	1.431 (2)	C5—H5	0.9500
S—N2	1.634 (2)	C6—C11	1.393 (3)
S—C6	1.779 (2)	C6—C7	1.393 (3)
O3—N3	1.216 (3)	C7—C8	1.387 (3)
O4—N3	1.214 (3)	C7—H7	0.9500
N1—C3	1.338 (3)	C8—C9	1.368 (3)
N1—C4	1.353 (3)	C8—H8	0.9500
N1—H1N	0.86 (3)	C9—C10	1.385 (3)
N2—C1	1.386 (2)	C9—H9	0.9500
N2—H2N	0.73 (3)	C10—C11	1.385 (3)
N3—C11	1.477 (3)	C10—H10	0.9500
C1—C2	1.395 (3)	Si—F3^{i}	1.6597 (14)
C1—C5	1.395 (3)	Si—F3	1.6597 (14)
C2—C3	1.367 (3)	Si—F2	1.6756 (13)
C2—H2	0.9500	Si—F2^{i}	1.6756 (13)
C3—H3	0.9500	Si—F1^{i}	1.7161 (13)
C4—C5	1.368 (3)	Si—F1	1.7161 (13)

O1—S—O2	120.30 (10)	C7—C6—S	116.81 (16)
O1—S—N2	109.10 (10)	C8—C7—C6	120.7 (2)
O2—S—N2	104.47 (10)	C8—C7—H7	119.7
O1—S—C6	105.83 (10)	C6—C7—H7	119.7
O2—S—C6	109.54 (10)	C9—C8—C7	120.8 (2)
N2—S—C6	106.98 (10)	C9—C8—H8	119.6
C3—N1—C4	122.20 (18)	C7—C8—H8	119.6
C3—N1—H1N	118 (2)	C8—C9—C10	119.9 (2)
C4—N1—H1N	120 (2)	C8—C9—H9	120.0
C1—N2—S	126.70 (16)	C10—C9—H9	120.0
C1—N2—H2N	122 (2)	C11—C10—C9	119.0 (2)
S—N2—H2N	110 (2)	C11—C10—H10	120.5
O4—N3—O3	123.2 (2)	C9—C10—H10	120.5
O4—N3—C11	117.7 (2)	C10—C11—C6	122.2 (2)
O3—N3—C11	119.09 (19)	C10—C11—N3	115.0 (2)
N2—C1—C2	122.01 (19)	C6—C11—N3	122.8 (2)
N2—C1—C5	118.34 (18)	F3^{i}—Si—F3	89.03 (10)
C2—C1—C5	119.64 (18)	F3^{i}—Si—F2	178.59 (7)
C3—C2—C1	118.97 (19)	F3—Si—F2	90.59 (7)
C3—C2—H2	120.5	F3^{i}—Si—F2^{i}	90.59 (7)
C1—C2—H2	120.5	F3—Si—F2^{i}	178.59 (7)
N1—C3—C2	120.30 (19)	F2—Si—F2^{i}	89.82 (9)
N1—C3—H3	119.8	F3^{i}—Si—F1^{i}	90.21 (7)
C2—C3—H3	119.8	F3—Si—F1^{i}	92.20 (7)
N1—C4—C5	119.82 (19)	F2—Si—F1^{i}	88.45 (7)
N1—C4—H4	120.1	F2^{i}—Si—F1^{i}	89.16 (7)
C5—C4—H4	120.1	F3^{i}—Si—F1	92.20 (7)
C4—C5—C1	119.02 (19)	F3—Si—F1	90.21 (7)
C4—C5—H5	120.5	F2—Si—F1	89.16 (7)
C1—C5—H5	120.5	F2^{i}—Si—F1	88.45 (7)
C11—C6—C7	117.3 (2)	F1^{i}—Si—F1	176.62 (9)
C11—C6—S	125.73 (17)		

O1—S—N2—C1	−65.8 (2)	O2—S—C6—C7	131.78 (16)
O2—S—N2—C1	164.31 (18)	N2—S—C6—C7	−115.54 (17)
C6—S—N2—C1	48.2 (2)	C11—C6—C7—C8	−1.3 (3)
S—N2—C1—C2	24.9 (3)	S—C6—C7—C8	−177.48 (17)
S—N2—C1—C5	−155.30 (16)	C6—C7—C8—C9	0.6 (3)
N2—C1—C2—C3	−178.80 (19)	C7—C8—C9—C10	0.4 (3)
C5—C1—C2—C3	1.4 (3)	C8—C9—C10—C11	−0.6 (3)
C4—N1—C3—C2	−1.5 (3)	C9—C10—C11—C6	−0.1 (3)
C1—C2—C3—N1	0.3 (3)	C9—C10—C11—N3	−178.4 (2)
C3—N1—C4—C5	0.9 (3)	C7—C6—C11—C10	1.0 (3)
N1—C4—C5—C1	0.7 (3)	S—C6—C11—C10	176.86 (17)
N2—C1—C5—C4	178.30 (18)	C7—C6—C11—N3	179.2 (2)
C2—C1—C5—C4	−1.9 (3)	S—C6—C11—N3	−5.0 (3)
O1—S—C6—C11	−175.15 (18)	O4—N3—C11—C10	−43.6 (3)
O2—S—C6—C11	−44.1 (2)	O3—N3—C11—C10	132.9 (2)

N2—S—C6—C11	68.6 (2)	O4—N3—C11—C6	138.1 (3)
O1—S—C6—C7	0.70 (18)	O3—N3—C11—C6	−45.4 (3)

Symmetry code: (i) −*x*+2, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···F1^{ii}	0.86 (3)	1.96 (3)	2.789 (2)	162 (3)
N2—H2*N*···F1^{iii}	0.73 (3)	1.97 (3)	2.690 (2)	171 (3)
C4—H4···F2^{ii}	0.95	2.35	3.141 (2)	141
C5—H5···F3^{iii}	0.95	2.50	3.426 (3)	165

Symmetry codes: (ii) −*x*+3/2, *y*+1/2, −*z*+3/2; (iii) −*x*+3/2, *y*−1/2, −*z*+3/2.