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1,1'-(*p*-Phenylene)bis(1*H*-imidazol-3-ium) hexafluoridosilicate(IV)

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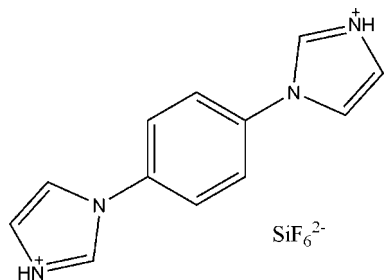
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.031; wR factor = 0.080; data-to-parameter ratio = 10.2.

In the title compound, $\text{C}_{12}\text{H}_{12}\text{N}_4^{2+} \cdot \text{SiF}_6^{2-}$, both the dication and the dianion are centrosymmetric. The dihedral angle between the 1*H*-imidazol-3-ium ring and the benzene ring is $27.80(11)^\circ$. An $\text{N}-\text{H} \cdots \text{F}$ hydrogen bond helps to establish the packing.

Related literature

For the amine synthesis, see: Cristau *et al.* (2004).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{12}\text{N}_4^{2+} \cdot \text{SiF}_6^{2-}$
 $M_r = 354.35$

Triclinic, $P\bar{1}$
 $a = 6.5608(13)$ Å

$b = 6.8060(14)$ Å
 $c = 8.7799(18)$ Å
 $\alpha = 85.88(3)^\circ$
 $\beta = 70.38(3)^\circ$
 $\gamma = 61.59(3)^\circ$
 $V = 322.97(17)$ Å³

$Z = 1$
Mo $K\alpha$ radiation
 $\mu = 0.26$ mm⁻¹
 $T = 293(2)$ K
 $0.02 \times 0.02 \times 0.01$ mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.836$, $T_{\max} = 1.000$
(expected range = 0.834–0.997)

3116 measured reflections
1119 independent reflections
894 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.035$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.080$
 $S = 1.02$
1119 reflections
110 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.27$ e Å⁻³
 $\Delta\rho_{\min} = -0.30$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N1}-\text{H1A} \cdots \text{F3}^i$	0.91 (3)	1.81 (3)	2.692 (2)	164 (3)

Symmetry code: (i) $x - 1, y, z$.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1997); software used to prepare material for publication: SHELXTL.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2649).

References

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supplementary materials

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1,1'-(*p*-Phenylene)bis(1*H*-imidazol-3-ium) hexafluorosilicate(IV)

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Comment

The title compound, (I), (Fig. 1) was obtained unexpectedly as the product of an attempted synthesis of a network complex of Mn(IV) using methanol and chloroform as the solvent. The dihedral angle between the 1*H*-imidazol-3-ium ring and the benzene ring of the cation is 27.80 (11)°.

An N—H···F hydrogen bond (Table 1) helps to establish the packing (Fig. 2), as well as electrostatic interactions.

Experimental

The ligand 1,4-di(1*H*-imidazol-1-yl)benzene was prepared according to the method of Cristau *et al.* (2004) from imidazole and 1,4-dibromobenzene. A buffer layer of a solution (8 ml) of methanol and chloroform (1:1) was carefully layered over the chloroform solution of the ligand (0.05 mmol, 6 ml). Then a methanol solution of Mn(SiF₆)₂ (0.05 mmol, 6 ml) was layered over the buffer layer. Colourless plates of (I) appeared at the boundary between methanol and chloroform after two weeks at room temperature.

Refinement

The C-bound H atoms were positioned geometrically and refined in the riding-model approximation, with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$.

The N-bound H atom was located in a difference map and its position was freely refined with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$.

Figures

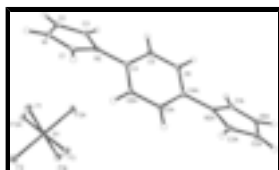


Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radius. Atoms with suffix A in the cation and anion are generated by the symmetry operations $(1 - x, 1 - y, z)$ and $(1 - x, -y, 1 - z)$.

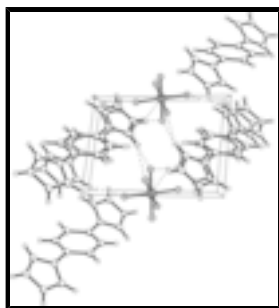


Fig. 2. The crystal packing for (I).

1,1'-(*p*-Phenylene)bis(1*H*-imidazol-3-ium) hexafluorosilicate(IV)

Crystal data

$C_{12}H_{12}N_4^{2+} \cdot Si_1F_6^{2-}$	$Z = 1$
$M_r = 354.35$	$F_{000} = 180$
Triclinic, $P\bar{1}$	$D_x = 1.822 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 6.5608 (13) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 6.8060 (14) \text{ \AA}$	Cell parameters from 869 reflections
$c = 8.7799 (18) \text{ \AA}$	$\theta = 2.5\text{--}27.9^\circ$
$\alpha = 85.88 (3)^\circ$	$\mu = 0.26 \text{ mm}^{-1}$
$\beta = 70.38 (3)^\circ$	$T = 293 (2) \text{ K}$
$\gamma = 61.59 (3)^\circ$	Block, colourless
$V = 322.97 (17) \text{ \AA}^3$	$0.02 \times 0.02 \times 0.01 \text{ mm}$

Data collection

Bruker SMART 1000 CCD diffractometer	1119 independent reflections
Radiation source: fine-focus sealed tube	894 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.035$
Detector resolution: 9 pixels mm^{-1}	$\theta_{\text{max}} = 25.0^\circ$
$T = 293(2) \text{ K}$	$\theta_{\text{min}} = 2.5^\circ$
ω scans	$h = -7 \rightarrow 7$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$k = -6 \rightarrow 8$
$T_{\text{min}} = 0.836$, $T_{\text{max}} = 1.000$	$l = -10 \rightarrow 10$
3116 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.031$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.080$	$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
1119 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
110 parameters	$\Delta\rho_{\text{max}} = 0.27 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.30 \text{ e \AA}^{-3}$
	Extinction correction: none

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 $F^2 > 2\sigma(F^2)$ 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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Si1	0.5000	0.0000	0.5000	0.0108 (2)
F3	0.50781 (19)	0.24334 (18)	0.52554 (12)	0.0157 (3)
F2	0.4698 (2)	-0.03743 (19)	0.69557 (12)	0.0180 (3)
F1	0.19141 (19)	0.14648 (18)	0.55382 (13)	0.0170 (3)
N1	-0.0959 (3)	0.2524 (3)	0.30612 (19)	0.0134 (4)
N2	0.1761 (3)	0.3241 (3)	0.13441 (18)	0.0109 (4)
C5	0.4441 (3)	0.4009 (3)	-0.1021 (2)	0.0125 (4)
H5	0.4065	0.3334	-0.1694	0.015*
C6	0.6031 (3)	0.4874 (3)	-0.1677 (2)	0.0125 (4)
H6	0.6720	0.4806	-0.2799	0.015*
C4	0.3405 (3)	0.4160 (3)	0.0661 (2)	0.0111 (4)
C1	0.0019 (3)	0.3849 (3)	0.2819 (2)	0.0130 (4)
H1	-0.0423	0.5024	0.3552	0.016*
C3	0.1846 (3)	0.1430 (3)	0.0630 (2)	0.0126 (4)
H3	0.2877	0.0672	-0.0399	0.015*
C2	0.0149 (3)	0.0993 (3)	0.1719 (2)	0.0138 (4)
H2	-0.0210	-0.0138	0.1587	0.017*
H1A	-0.212 (5)	0.239 (5)	0.392 (3)	0.051 (8)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0114 (4)	0.0117 (4)	0.0090 (4)	-0.0065 (3)	-0.0016 (3)	-0.0002 (3)
F3	0.0152 (6)	0.0141 (6)	0.0162 (6)	-0.0092 (5)	0.0005 (5)	-0.0025 (4)
F2	0.0231 (7)	0.0216 (7)	0.0101 (6)	-0.0123 (5)	-0.0042 (5)	0.0010 (5)
F1	0.0128 (6)	0.0174 (6)	0.0191 (6)	-0.0069 (5)	-0.0033 (5)	-0.0015 (5)
N1	0.0121 (9)	0.0175 (9)	0.0127 (9)	-0.0096 (7)	-0.0030 (7)	0.0026 (7)
N2	0.0099 (8)	0.0121 (9)	0.0103 (8)	-0.0053 (7)	-0.0028 (6)	0.0004 (6)
C5	0.0150 (10)	0.0118 (10)	0.0129 (10)	-0.0069 (8)	-0.0065 (8)	0.0012 (7)
C6	0.0126 (10)	0.0136 (10)	0.0088 (9)	-0.0050 (8)	-0.0024 (8)	0.0001 (7)
C4	0.0080 (9)	0.0083 (10)	0.0142 (9)	-0.0029 (8)	-0.0021 (7)	0.0015 (7)
C1	0.0109 (10)	0.0157 (10)	0.0111 (9)	-0.0054 (8)	-0.0036 (8)	0.0002 (7)

supplementary materials

C3	0.0113 (10)	0.0116 (10)	0.0139 (9)	-0.0051 (8)	-0.0030 (8)	-0.0027 (8)
C2	0.0144 (10)	0.0123 (10)	0.0167 (10)	-0.0073 (8)	-0.0058 (8)	0.0001 (8)

Geometric parameters (Å, °)

Si1—F2 ⁱ	1.6761 (11)	N2—C4	1.438 (2)
Si1—F2	1.6761 (11)	C5—C6	1.378 (2)
Si1—F1	1.6795 (12)	C5—C4	1.391 (2)
Si1—F1 ⁱ	1.6795 (13)	C5—H5	0.9300
Si1—F3	1.7140 (11)	C6—C4 ⁱⁱ	1.379 (3)
Si1—F3 ⁱ	1.7140 (11)	C6—H6	0.9300
N1—C1	1.305 (2)	C4—C6 ⁱⁱ	1.379 (3)
N1—C2	1.372 (2)	C1—H1	0.9300
N1—H1A	0.91 (2)	C3—C2	1.342 (3)
N2—C1	1.337 (2)	C3—H3	0.9300
N2—C3	1.392 (2)	C2—H2	0.9300
F2 ⁱ —Si1—F2	180.0	C3—N2—C4	125.57 (15)
F2 ⁱ —Si1—F1	90.51 (7)	C6—C5—C4	119.04 (17)
F2—Si1—F1	89.49 (7)	C6—C5—H5	120.5
F2 ⁱ —Si1—F1 ⁱ	89.49 (7)	C4—C5—H5	120.5
F2—Si1—F1 ⁱ	90.51 (7)	C5—C6—C4 ⁱⁱ	119.68 (17)
F1—Si1—F1 ⁱ	180.0	C5—C6—H6	120.2
F2 ⁱ —Si1—F3	90.12 (6)	C4 ⁱⁱ —C6—H6	120.2
F2—Si1—F3	89.88 (6)	C6 ⁱⁱ —C4—C5	121.27 (17)
F1—Si1—F3	89.79 (6)	C6 ⁱⁱ —C4—N2	119.73 (16)
F1 ⁱ —Si1—F3	90.21 (6)	C5—C4—N2	118.99 (16)
F2 ⁱ —Si1—F3 ⁱ	89.88 (6)	N1—C1—N2	108.74 (17)
F2—Si1—F3 ⁱ	90.12 (6)	N1—C1—H1	125.6
F1—Si1—F3 ⁱ	90.21 (6)	N2—C1—H1	125.6
F1 ⁱ —Si1—F3 ⁱ	89.79 (6)	C2—C3—N2	106.52 (16)
F3—Si1—F3 ⁱ	180.0	C2—C3—H3	126.7
C1—N1—C2	109.41 (16)	N2—C3—H3	126.7
C1—N1—H1A	133.6 (18)	C3—C2—N1	107.38 (17)
C2—N1—H1A	116.9 (18)	C3—C2—H2	126.3
C1—N2—C3	107.95 (15)	N1—C2—H2	126.3
C1—N2—C4	126.30 (16)		
C4—C5—C6—C4 ⁱⁱ	0.9 (3)	C2—N1—C1—N2	-0.1 (2)
C6—C5—C4—C6 ⁱⁱ	-0.9 (3)	C3—N2—C1—N1	0.4 (2)
C6—C5—C4—N2	-179.50 (17)	C4—N2—C1—N1	-174.84 (16)
C1—N2—C4—C6 ⁱⁱ	25.2 (3)	C1—N2—C3—C2	-0.6 (2)
C3—N2—C4—C6 ⁱⁱ	-149.18 (18)	C4—N2—C3—C2	174.70 (16)
C1—N2—C4—C5	-156.19 (18)	N2—C3—C2—N1	0.5 (2)
C3—N2—C4—C5	29.4 (3)	C1—N1—C2—C3	-0.3 (2)

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

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—H1A···F3 ⁱⁱⁱ	0.91 (3)	1.81 (3)	2.692 (2)	164 (3)

Symmetry codes: (iii) $x-1, y, z$.

Fig. 1

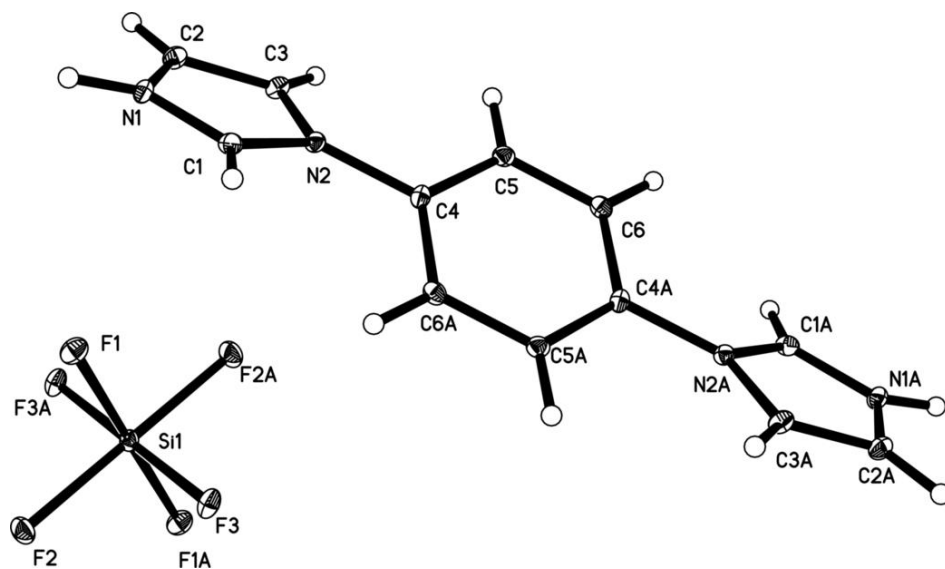


Fig. 2

