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Tris(ethylenediamine)zinc(II) hexafluoridosilicate

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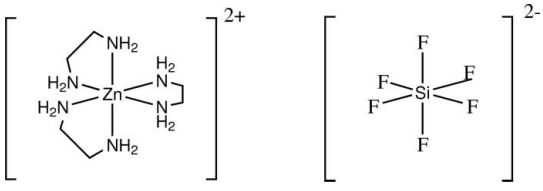
Key indicators: single-crystal X-ray study; T = 93 K; mean σ(C–C) = 0.003 Å; R factor = 0.027; wR factor = 0.059; data-to-parameter ratio = 16.7.

The title compound, $[Zn(C_2H_8N_2)_3](SiF_6)$, was synthesized ionothermally using choline chloride–imidazolidone as solvent and template provider. In the crystal structure, the anions and cations are located on special positions of site symmetry 3.2 and show a typical octahedral geometry. The Zn^{II} ion is coordinated by six N atoms from three ethylenediamine molecules. The crystal structure displays weak hydrogen bonding between $[SiF_6]^{2-}$ anions and the ethylenediamine NH hydrogen atoms.

Related literature

For related structures, see: Ray *et al.* (1973); Bernhardt & Riley (2003); Cernak *et al.* (1984); Emsley *et al.* (1989); Cheng *et al.* (2008).



Experimental

Crystal data

$[Zn(C_2H_8N_2)_3](SiF_6)$
M_r = 387.77
Hexagonal, $P6_522$
a = 9.192 (2) Å
c = 9.755 (3) Å
V = 713.8 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.87 mm^{-1}
T = 93 K
0.10 × 0.10 × 0.10 mm

Data collection

Rigaku Mercury CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2004)
T_{min} = 0.835, T_{max} = 0.835
4809 measured reflections
534 independent reflections
499 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.059
S = 1.11
534 reflections
32 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Absolute structure: Flack (1983), 177 Friedel pairs
Flack parameter: 0.01 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···F1^{i}	0.92	2.26	3.113 (3)	155
N1—H1A···F1^{ii}	0.92	2.49	3.239 (3)	139
N1—H1B···F1^{iii}	0.92	2.25	3.153 (3)	166

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

Data collection: *CrystalClear* (Rigaku, 2004); 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: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5123).

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supplementary materials

Acta Cryst. (2009). E**65**, m1522 [**doi:10.1107/S1600536809045693**]

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Comment

A large number of salts with the general formula MG_6LR_6, where *M* is a bivalent metal, G may be water or ammonia, *L* is a quadrivalent element like Si, Sn, Ti or Zr, and *R* may be Cl, F or CN, (Ray *et al.*, 1973), were studied. We report a similar type of the title salt containing organic molecules. The molecule of the title salt, shown in Fig. 1, consists of one $Zn(C_2N_2H_8)_3$ cation and one SiF_6 anion. The coordination of ZnII centers through bridging-bidentate ethylenediamine groups forms a wind-stick-like cluster. The $Zn(C_2N_2H_8)_3$ cluster and SiF_6 octahedra are stacked alternately along the threefold axis in approximately CsCl-type packing.

Experimental

A typical synthetic procedure for $Zn(C_2N_2H_8)_3.SiF_6$ was as follows: a Teflon-lined autoclave (volume 15 ml) was charged with the ionic liquid [composed of choline chloride (1630 mg, 11.4 mmol) and imidazolidone (2.045 g, 22.8 mmol)], zinc acetate (168 mg, 0.74 mmol), NH_4F (71 mg, 1.85 mmol), and silica (49 mg, 0.74 mmol) and heated in an oven at 180 °C for 3 days. Ethylenediamine($C_2N_2H_8$), derived from decomposition of the imidazolidone component of the deep-eutectic solvent (DES) itself, is delivered to the synthesis. The synthesized samples were washed with distilled water in an ultrasonic bath, then washed with acetone, and dried at room temperature in air. The colorless crystals of the title salt were abtained with suitable size for single-crystal X-ray analysis.

Refinement

All H atoms were fixed geometrically (C—H = 0.99 Å, N—H = 0.92 Å) and treated as riding with U_{iso}(H) = 1.2Ueq of the parent atom.

Figures

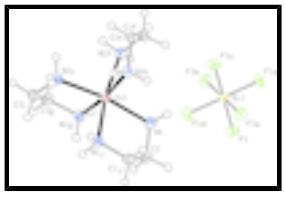


Fig. 1. The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

Tris(ethylenediamine)zinc(II) hexafluoridosilicate

Crystal data

$[Zn(C_2H_8N_2)_3](SiF_6)$ $Z = 2$

M_r = 387.77	F_{000} = 400
Trigonal, $P6_322$	D_x = 1.804 Mg m^{-3}
Hall symbol: P 6c 2c	Mo $K\alpha$ radiation, λ = 0.71073 Å
a = 9.192 (2) Å	Cell parameters from 1402 reflections
b = 9.192 (2) Å	θ = 6.6–54.6º
c = 9.755 (3) Å	μ = 1.87 mm^{-1}
α = 90º	T = 93 K
β = 90º	Prism, colorless
γ = 120º	0.10 × 0.10 × 0.10 mm
V = 713.8 (3) Å^3	

Data collection

Rigaku Mercury CCD diffractometer	534 independent reflections
Radiation source: rotating anode	499 reflections with $I > 2\sigma(I)$
Monochromator: confocal	R_{int} = 0.045
Detector resolution: 0.83 pixels mm^{-1}	θ_{max} = 27.4º
T = 93 K	θ_{min} = 3.3º
ω scans	h = −10→11
Absorption correction: Multi-scan (CrystalClear; Rigaku, 2004)	k = −10→11
T_{min} = 0.835, T_{max} = 0.835	l = −11→9
4809 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.027	$w = 1/[\sigma^2(F_o^2) + (0.0158P)^2 + 0.5912P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.059	$(\Delta/\sigma)_{max} < 0.001$
S = 1.11	$\Delta\rho_{max}$ = 0.51 e Å^{-3}
534 reflections	$\Delta\rho_{min}$ = −0.38 e Å^{-3}
32 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 177 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.01 (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}
Si1	0.3333	0.6667	0.7500	0.0135 (3)
F1	0.48288 (18)	0.6657 (2)	0.85081 (14)	0.0250 (3)
Zn1	0.6667	0.3333	0.7500	0.01508 (18)
N1	0.8544 (2)	0.5434 (2)	0.87149 (19)	0.0191 (4)
H1A	0.8277	0.5243	0.9631	0.023*
H1B	0.9584	0.5539	0.8589	0.023*
C1	0.8584 (4)	0.6997 (3)	0.8277 (2)	0.0222 (5)
H2A	0.9655	0.7986	0.8566	0.027*
H2B	0.7651	0.7070	0.8716	0.027*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0151 (4)	0.0151 (4)	0.0102 (7)	0.0076 (2)	0.000	0.000
F1	0.0260 (7)	0.0352 (8)	0.0183 (7)	0.0186 (7)	−0.0057 (6)	−0.0011 (7)
Zn1	0.0169 (2)	0.0169 (2)	0.0115 (3)	0.00843 (11)	0.000	0.000
N1	0.0185 (10)	0.0240 (11)	0.0137 (11)	0.0097 (9)	−0.0018 (8)	0.0003 (8)
C1	0.0234 (13)	0.0195 (11)	0.0225 (12)	0.0099 (13)	−0.0031 (12)	−0.0037 (8)

Geometric parameters (Å, °)

Si1—F1^{i}	1.6938 (13)	Zn1—N1^{v}	2.186 (2)
Si1—F1^{ii}	1.6938 (13)	Zn1—N1^{viii}	2.1863 (19)
Si1—F1^{iii}	1.6938 (14)	Zn1—N1^{ix}	2.1863 (19)
Si1—F1^{iv}	1.6938 (14)	N1—C1	1.482 (3)
Si1—F1	1.6938 (13)	N1—H1A	0.9200
Si1—F1^{v}	1.6938 (13)	N1—H1B	0.9200
Zn1—N1^{vi}	2.1863 (19)	C1—C1^{viii}	1.523 (4)
Zn1—N1^{vii}	2.1863 (19)	C1—H2A	0.9900
Zn1—N1	2.186 (2)	C1—H2B	0.9900
F1^{i}—Si1—F1^{ii}	90.69 (10)	N1^{vi}—Zn1—N1^{viii}	93.40 (7)
F1^{i}—Si1—F1^{iii}	89.68 (7)	N1^{vii}—Zn1—N1^{viii}	170.67 (11)
F1^{ii}—Si1—F1^{iii}	89.95 (10)	N1—Zn1—N1^{viii}	80.19 (10)
F1^{i}—Si1—F1^{iv}	89.95 (10)	N1^{v}—Zn1—N1^{viii}	93.40 (7)
F1^{ii}—Si1—F1^{iv}	89.68 (7)	N1^{vi}—Zn1—N1^{ix}	170.67 (11)
F1^{iii}—Si1—F1^{iv}	179.47 (11)	N1^{vii}—Zn1—N1^{ix}	93.40 (7)
F1^{i}—Si1—F1	179.47 (11)	N1—Zn1—N1^{ix}	93.40 (7)

F1^{ii}—Si1—F1	89.68 (7)	N1^{v}—Zn1—N1^{ix}	80.19 (10)
F1^{iii}—Si1—F1	90.69 (10)	N1^{viii}—Zn1—N1^{ix}	93.74 (11)
F1^{iv}—Si1—F1	89.68 (7)	C1—N1—Zn1	109.01 (14)
F1^{i}—Si1—F1^{v}	89.68 (7)	C1—N1—H1A	109.9
F1^{ii}—Si1—F1^{v}	179.47 (11)	Zn1—N1—H1A	109.9
F1^{iii}—Si1—F1^{v}	89.68 (7)	C1—N1—H1B	109.9
F1^{iv}—Si1—F1^{v}	90.69 (10)	Zn1—N1—H1B	109.9
F1—Si1—F1^{v}	89.95 (10)	H1A—N1—H1B	108.3
N1^{vi}—Zn1—N1^{vii}	80.19 (10)	N1—C1—C1^{viii}	109.52 (17)
N1^{vi}—Zn1—N1	93.74 (11)	N1—C1—H2A	109.8
N1^{vii}—Zn1—N1	93.40 (7)	C1^{viii}—C1—H2A	109.8
N1^{vi}—Zn1—N1^{v}	93.40 (7)	N1—C1—H2B	109.8
N1^{vii}—Zn1—N1^{v}	93.74 (11)	C1^{viii}—C1—H2B	109.8
N1—Zn1—N1^{v}	170.67 (11)	H2A—C1—H2B	108.2
N1^{vi}—Zn1—N1—C1	106.99 (17)	N1^{ix}—Zn1—N1—C1	−79.03 (19)
N1^{vii}—Zn1—N1—C1	−172.64 (16)	Zn1—N1—C1—C1^{viii}	−39.9 (3)
N1^{viii}—Zn1—N1—C1	14.19 (13)		

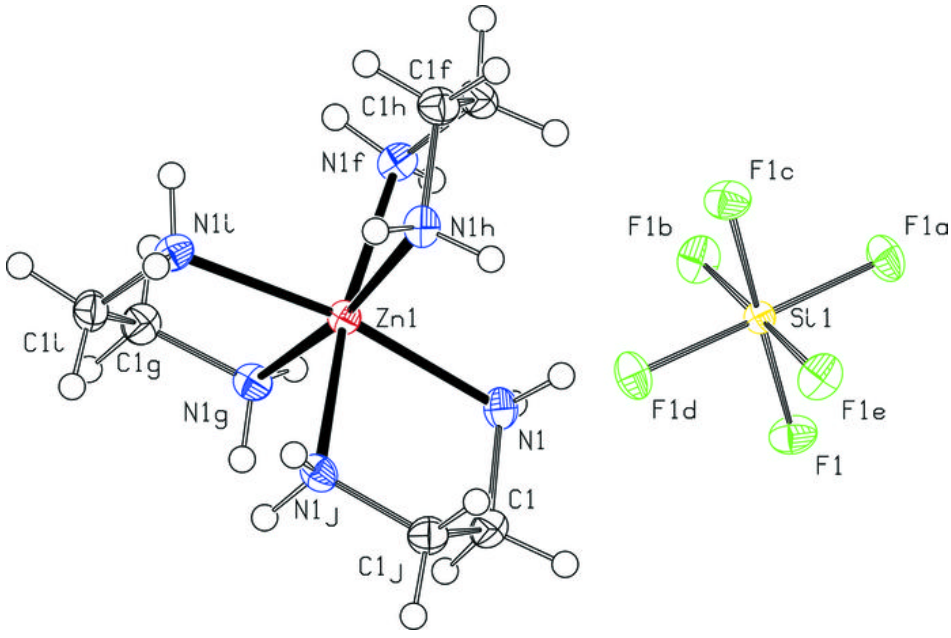
Symmetry codes: (i) −*x*+*y*, *y*, −*z*+3/2; (ii) −*y*+1, *x*−*y*+1, *z*; (iii) *x*, *x*−*y*+1, −*z*+3/2; (iv) −*x*+*y*, −*x*+1, *z*; (v) −*y*+1, −*x*+1, −*z*+3/2; (vi) *x*, *x*−*y*, −*z*+3/2; (vii) −*x*+*y*+1, −*x*+1, *z*; (viii) −*x*+*y*+1, *y*, −*z*+3/2; (ix) −*y*+1, *x*−*y*, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···F1^{x}	0.92	2.26	3.113 (3)	155
N1—H1A···F1^{xi}	0.92	2.49	3.239 (3)	139
N1—H1B···F1^{vii}	0.92	2.25	3.153 (3)	166

Symmetry codes: (x) *y*, *x*, −*z*+2; (xi) *x*−*y*+1, −*y*+1, −*z*+2; (vii) −*x*+*y*+1, −*x*+1, *z*.

Fig. 1


C1f
C1h
N1f
N1i
N1h
Zn1
C1i
C1g
N1g
N1
N1j
C1
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F1c
F1b
F1a
Si1
F1d
F1e
F1