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Hexaaquahexakis(μ_2 -3,5-diamino-4H-1,2,4-triazole)trinickel(II) tris(hexafluoridosilicate) icosahydrate

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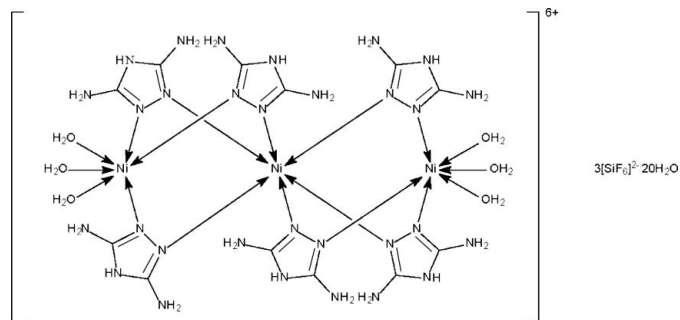
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{Si}-\text{F}) = 0.003$ Å; disorder in solvent or counterion; R factor = 0.033; wR factor = 0.107; data-to-parameter ratio = 13.1.

The trinuclear cation of the title compound, $[\text{Ni}_3(\text{C}_2\text{H}_5\text{N}_3)_6(\text{H}_2\text{O})_6][\text{SiF}_6]_3 \cdot 20\text{H}_2\text{O}$, has the six 3,5-diamino-1,2,4-triazole ligands each bridging two metal atoms; the metal atom in the middle, which lies on a special position (of 32 site symmetry), is connected to six N atoms in an octahedral geometry. The other metal atom, which lies on a special position (of 3 site symmetry), is connected to three N atoms and three O atoms. One hexafluoridosilicate anion lies on a site of 3 symmetry and the other lies on a site of $\bar{3}$ symmetry. The hexacation, dianions and uncoordinated water molecules interact through hydrogen bonds to form a three-dimensional network. One uncoordinated water molecule is disordered, with site occupancy 0.3.

Related literature

For the structure of the title hexacation as the hydrated sulfate salt, see: Zhang *et al.* (2007).



Experimental

Crystal data

$[\text{Ni}_3(\text{C}_2\text{H}_5\text{N}_3)_6(\text{H}_2\text{O})_6][\text{SiF}_6]_3 \cdot 20\text{H}_2\text{O}$
 $V = 9469.8$ (9) Å³
 $Z = 6$
 Mo $K\alpha$ radiation
 $\mu = 1.09$ mm⁻¹
 $T = 293$ (2) K
 $0.49 \times 0.46 \times 0.44$ mm

Data collection

Bruker APEX area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\text{min}} = 0.560$, $T_{\text{max}} = 0.646$
 25347 measured reflections
 2420 independent reflections
 2093 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.033$
 $wR(F^2) = 0.106$
 $S = 1.06$
 2420 reflections
 185 parameters
 23 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.40$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.42$ e Å⁻³

Table 1

Selected bond lengths (Å).

Ni1—N1	2.062 (2)	Ni2—N2	2.111 (2)
Ni1—O1	2.104 (2)		

Data collection: SMART (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: publCIF (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2084).

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supplementary materials

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Hexaaquahexakis(μ_2 -3,5-diamino-4*H*-1,2,4-triazole)trinickel(II) tris(hexafluorosilicate) icosahydrate

L.-P. Wu, S.-M. Zhao, G.-F. Zhang and S. W. Ng

Comment

A recent study reported the nickel sulfate complex of 3,5-diamino-1,2,4-triazole. The cation is a centrosymmetric trinuclear hexacation $[\text{Ni}_3(\text{C}_2\text{H}_5\text{N}_5)_6(\text{H}_2\text{O})_6]^{6+}$, whose charge is balanced by the sulfate anions. The *N*-heterocyclic ligands each bridge two nickel atoms (Zhang *et al.*, 2007). The corresponding synthesis with nickel hexafluorosilicate in place of nickel sulfate gave the analogous cluster cation (Scheme I, Fig. 1). The cation and anions interact through the coordinated and free water molecules to give rise to a three-dimensional, hydrogen bonded network.

Experimental

Single crystal of the compound were grown by diffusing 3,5-diamino-1,2,4-triazole (0.020 g, 0.2 mmol) dissolved in methanol (5 ml) into nickel(II) hexafluorosilicate (0.027 g, 0.1 mmol) dissolved in water (5 ml).

Refinement

As one of the five water molecules (O5) lies on a special position of 2 site symmetry, the occupancy would be 0.5. However, the refinement of this atom at the default occupancy lead to a large temperature factor. The refinement of the occupancy factor led to a value of about 0.3; this atom was then allowed to refine off the symmetry element. As the occupancy was nearly 0.3, the occupancy was arbitrarily fixed as 0.3333 so that the formula unit has 20 lattice water molecules.

The N- and O-bound H atoms (other than those of the disordered water molecule) were found in difference maps and were refined with distance restraints of $\text{O}-\text{H} = \text{N}-\text{H} = 0.85 \pm 0.01 \text{ \AA}$; for the water molecules, an additional $\text{H}\cdots\text{H} = 1.39 \pm 0.01 \text{ \AA}$ restraint was imposed. The $U_{\text{iso}}(\text{H})$ values were tied to those of the parent atoms by a factor of 1.5. The H atoms of the disordered water molecule were placed in chemically sensible positions but were not refined.

Figures

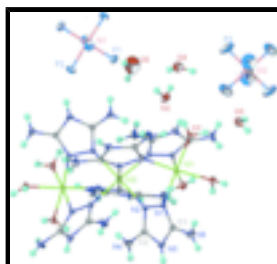


Fig. 1. Thermal ellipsoid plot of the title compound. Displacement ellipsoids are drawn at the 25% probability level.

Hexaaquahexakis(μ_2 -3,5-diamino-4H-1,2,4-triazole)trinickel(II) tris(hexafluorosilicate) icosahydrate

Crystal data

$[\text{Ni}_3(\text{C}_2\text{H}_5\text{N}_3)_6(\text{H}_2\text{O})_6][\text{SiF}_6]_3 \cdot 20\text{H}_2\text{O}$	$Z = 6$
$M_r = 1665.48$	$F_{000} = 5160$
Trigonal, $R\bar{3}c$	$D_x = 1.752 \text{ Mg m}^{-3}$
Hall symbol: $-R\ 3\ 2''c$	Mo $K\alpha$ radiation
$a = 13.024 (1) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 13.024 \text{ \AA}$	Cell parameters from 5839 reflections
$c = 64.462 (5) \text{ \AA}$	$\theta = 3.1\text{--}28.2^\circ$
$\alpha = 90^\circ$	$\mu = 1.09 \text{ mm}^{-1}$
$\beta = 90^\circ$	$T = 293 (2) \text{ K}$
$\gamma = 120^\circ$	Block, blue
$V = 9469.8 (9) \text{ \AA}^3$	$0.49 \times 0.46 \times 0.44 \text{ mm}$

Data collection

Bruker APEX area-detector diffractometer	2420 independent reflections
Radiation source: fine-focus sealed tube	2093 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.021$
$T = 293(2) \text{ K}$	$\theta_{\text{max}} = 27.5^\circ$
φ and ω scans	$\theta_{\text{min}} = 3.1^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -16 \rightarrow 16$
$T_{\text{min}} = 0.560$, $T_{\text{max}} = 0.646$	$k = -16 \rightarrow 16$
25347 measured reflections	$l = -79 \rightarrow 83$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.033$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.106$	$w = 1/[\sigma^2(F_o^2) + (0.0612P)^2 + 17.0807P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
2420 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
185 parameters	$\Delta\rho_{\text{max}} = 0.40 \text{ e \AA}^{-3}$
23 restraints	$\Delta\rho_{\text{min}} = -0.42 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Ni1	0.6667	0.3333	0.140787 (6)	0.02689 (14)	
Ni2	0.6667	0.3333	0.0833	0.02377 (16)	
Si1	1.0000	0.0000	0.083802 (16)	0.0331 (2)	
Si2	0.3333	-0.3333	0.1667	0.0552 (5)	
F1	0.89685 (14)	0.00226 (15)	0.09960 (2)	0.0623 (4)	
F2	1.00168 (16)	0.10635 (14)	0.06943 (3)	0.0648 (4)	
F3	0.3983 (3)	-0.3868 (3)	0.15169 (5)	0.1399 (12)	
O1	0.72286 (15)	0.24063 (14)	0.16010 (2)	0.0398 (3)	
H11	0.723 (3)	0.1843 (18)	0.1533 (3)	0.060*	
H12	0.699 (3)	0.217 (2)	0.1724 (2)	0.060*	
O2	0.7536 (2)	0.0854 (2)	0.13625 (4)	0.0660 (5)	
H21	0.713 (2)	0.0108 (10)	0.1358 (6)	0.099*	
H22	0.8267 (10)	0.108 (3)	0.1361 (7)	0.099*	
O3	0.65459 (19)	0.15616 (17)	0.19955 (3)	0.0599 (5)	
H31	0.5874 (17)	0.094 (2)	0.1978 (5)	0.090*	
H32	0.650 (3)	0.197 (2)	0.2092 (4)	0.090*	
O4	0.6041 (2)	-0.1661 (3)	0.12859 (4)	0.0796 (6)	
H41	0.576 (3)	-0.184 (4)	0.1164 (3)	0.119*	
H42	0.551 (3)	-0.182 (4)	0.1374 (4)	0.119*	
O5	0.664 (3)	-0.1549 (17)	0.0893 (3)	0.185 (7)	0.3333
H51	0.7382	-0.1072	0.0905	0.278*	0.3333
H52	0.6338	-0.1239	0.0816	0.278*	0.3333
N1	0.61588 (14)	0.43372 (14)	0.12352 (2)	0.0290 (3)	
N2	0.59282 (14)	0.41343 (14)	0.10195 (2)	0.0293 (3)	
N3	0.52872 (17)	0.53100 (17)	0.11289 (3)	0.0381 (4)	
H3	0.500 (2)	0.576 (2)	0.1117 (4)	0.057*	
N4	0.5100 (2)	0.4865 (2)	0.07683 (3)	0.0508 (5)	
H4A	0.514 (3)	0.449 (3)	0.0663 (4)	0.076*	
H4B	0.468 (2)	0.517 (3)	0.0744 (6)	0.076*	
N5	0.5810 (2)	0.5478 (2)	0.14862 (3)	0.0556 (6)	
H5A	0.625 (3)	0.540 (3)	0.1574 (4)	0.083*	
H5B	0.535 (3)	0.575 (3)	0.1509 (5)	0.083*	
C1	0.57611 (18)	0.50413 (18)	0.12943 (3)	0.0343 (4)	
C2	0.54160 (17)	0.47436 (17)	0.09630 (3)	0.0336 (4)	

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.02997 (17)	0.02997 (17)	0.0207 (2)	0.01499 (9)	0.000	0.000
Ni2	0.0253 (2)	0.0253 (2)	0.0207 (3)	0.01265 (10)	0.000	0.000
Si1	0.0300 (3)	0.0300 (3)	0.0394 (5)	0.01500 (15)	0.000	0.000
Si2	0.0333 (5)	0.0333 (5)	0.0992 (15)	0.0166 (2)	0.000	0.000
F1	0.0555 (9)	0.0683 (10)	0.0701 (10)	0.0363 (8)	0.0170 (7)	0.0016 (8)
F2	0.0710 (10)	0.0574 (9)	0.0741 (10)	0.0382 (8)	-0.0004 (8)	0.0199 (8)

supplementary materials

F3	0.146 (3)	0.176 (3)	0.163 (3)	0.129 (3)	0.0160 (19)	-0.025 (2)
O1	0.0497 (8)	0.0454 (8)	0.0292 (7)	0.0274 (7)	-0.0020 (6)	0.0041 (6)
O2	0.0624 (12)	0.0722 (13)	0.0750 (13)	0.0424 (11)	-0.0019 (10)	-0.0133 (11)
O3	0.0764 (13)	0.0541 (11)	0.0436 (9)	0.0283 (10)	0.0132 (9)	-0.0001 (8)
O4	0.0709 (15)	0.0812 (15)	0.0871 (16)	0.0383 (13)	-0.0030 (12)	-0.0025 (14)
O5	0.119 (7)	0.216 (10)	0.178 (13)	0.051 (7)	-0.015 (9)	-0.010 (8)
N1	0.0349 (8)	0.0329 (8)	0.0226 (7)	0.0194 (6)	0.0006 (6)	-0.0010 (6)
N2	0.0352 (8)	0.0355 (8)	0.0219 (7)	0.0213 (7)	-0.0009 (6)	-0.0005 (6)
N3	0.0476 (10)	0.0440 (10)	0.0366 (9)	0.0334 (8)	-0.0010 (7)	-0.0015 (7)
N4	0.0743 (15)	0.0720 (14)	0.0343 (10)	0.0578 (13)	-0.0109 (9)	-0.0043 (9)
N5	0.0821 (16)	0.0803 (15)	0.0348 (10)	0.0635 (14)	-0.0071 (10)	-0.0157 (10)
C1	0.0390 (10)	0.0367 (10)	0.0319 (9)	0.0224 (9)	0.0008 (8)	-0.0023 (8)
C2	0.0377 (10)	0.0374 (10)	0.0312 (9)	0.0229 (9)	-0.0009 (7)	0.0006 (7)

Geometric parameters (Å, °)

Ni1—N1	2.062 (2)	Si2—F3	1.649 (3)
Ni1—N1 ⁱ	2.062 (2)	O1—H11	0.86 (3)
Ni1—N1 ⁱⁱ	2.062 (2)	O1—H12	0.85 (3)
Ni1—O1	2.104 (2)	O2—H21	0.84 (3)
Ni1—O1 ⁱ	2.104 (2)	O2—H22	0.84 (3)
Ni1—O1 ⁱⁱ	2.104 (2)	O3—H31	0.85 (3)
Ni2—N2 ⁱⁱⁱ	2.111 (2)	O3—H32	0.84 (3)
Ni2—N2 ^{iv}	2.111 (2)	O4—H41	0.85 (3)
Ni2—N2 ^v	2.111 (2)	O4—H42	0.84 (3)
Ni2—N2 ⁱⁱ	2.111 (2)	O5—H51	0.85
Ni2—N2 ⁱ	2.111 (2)	O5—H52	0.85
Ni2—N2	2.111 (2)	N1—C1	1.315 (2)
Si1—F2	1.6574 (15)	N1—N2	1.419 (2)
Si1—F2 ^{vi}	1.6574 (15)	N2—C2	1.318 (3)
Si1—F2 ^{vii}	1.6574 (15)	N3—C2	1.356 (3)
Si1—F1 ^{viii}	1.6976 (15)	N3—C1	1.362 (3)
Si1—F1 ^{vi}	1.6976 (15)	N3—H3	0.85 (1)
Si1—F1	1.6976 (15)	N4—C2	1.354 (3)
Si2—F3 ^{viii}	1.649 (3)	N4—H4A	0.85 (3)
Si2—F3 ^{ix}	1.649 (3)	N4—H4B	0.84 (3)
Si2—F3 ^x	1.649 (3)	N5—C1	1.350 (3)
Si2—F3 ^{xi}	1.649 (3)	N5—H5A	0.84 (3)
Si2—F3 ^{xii}	1.649 (3)	N5—H5B	0.85 (3)
N1 ⁱⁱ —Ni1—N1 ⁱ	93.61 (6)	F1 ^{vi} —Si1—F1	87.74 (9)
N1 ⁱⁱ —Ni1—N1	93.61 (6)	F3 ^{viii} —Si2—F3 ^{ix}	89.21 (17)
N1 ⁱ —Ni1—N1	93.61 (6)	F3 ^{viii} —Si2—F3 ^x	89.21 (17)
N1 ⁱⁱ —Ni1—O1	87.31 (6)	F3 ^{ix} —Si2—F3 ^x	89.21 (17)
N1 ⁱ —Ni1—O1	90.45 (6)	F3 ^{viii} —Si2—F3 ^{xi}	90.79 (17)

N1—Ni1—O1	175.77 (6)	F3 ^{ix} —Si2—F3 ^{xi}	90.79 (17)
N1 ⁱⁱ —Ni1—O1 ⁱ	90.45 (6)	F3 ^x —Si2—F3 ^{xi}	179.995 (1)
N1 ⁱ —Ni1—O1 ⁱ	175.77 (6)	F3 ^{viii} —Si2—F3 ^{xii}	90.79 (17)
N1—Ni1—O1 ⁱ	87.31 (6)	F3 ^{ix} —Si2—F3 ^{xii}	179.995 (1)
O1—Ni1—O1 ⁱ	88.56 (6)	F3 ^x —Si2—F3 ^{xii}	90.79 (17)
N1 ⁱⁱ —Ni1—O1 ⁱⁱ	175.78 (6)	F3 ^{xi} —Si2—F3 ^{xii}	89.21 (17)
N1 ⁱ —Ni1—O1 ⁱⁱ	87.31 (6)	F3 ^{viii} —Si2—F3	179.997 (1)
N1—Ni1—O1 ⁱⁱ	90.45 (6)	F3 ^{ix} —Si2—F3	90.79 (17)
O1—Ni1—O1 ⁱⁱ	88.56 (6)	F3 ^x —Si2—F3	90.79 (17)
O1 ⁱ —Ni1—O1 ⁱⁱ	88.56 (6)	F3 ^{xi} —Si2—F3	89.21 (17)
N2 ⁱⁱⁱ —Ni2—N2 ^{iv}	90.87 (6)	F3 ^{xii} —Si2—F3	89.21 (17)
N2 ⁱⁱⁱ —Ni2—N2 ^v	90.87 (6)	Ni1—O1—H11	110 (2)
N2 ^{iv} —Ni2—N2 ^v	90.87 (6)	Ni1—O1—H12	126 (2)
N2 ⁱⁱⁱ —Ni2—N2 ⁱⁱ	87.57 (8)	H11—O1—H12	109 (2)
N2 ^{iv} —Ni2—N2 ⁱⁱ	177.79 (8)	H21—O2—H22	111 (2)
N2 ^v —Ni2—N2 ⁱⁱ	90.72 (8)	H31—O3—H32	110 (2)
N2 ⁱⁱⁱ —Ni2—N2 ⁱ	90.72 (8)	H41—O4—H42	111 (2)
N2 ^{iv} —Ni2—N2 ⁱ	87.57 (8)	H51—O5—H52	109.4
N2 ^v —Ni2—N2 ⁱ	177.79 (8)	C1—N1—N2	107.04 (15)
N2 ⁱⁱ —Ni2—N2 ⁱ	90.88 (6)	C1—N1—Ni1	130.48 (12)
N2 ⁱⁱⁱ —Ni2—N2	177.79 (8)	N2—N1—Ni1	121.05 (11)
N2 ^{iv} —Ni2—N2	90.72 (8)	C2—N2—N1	106.38 (15)
N2 ^v —Ni2—N2	87.57 (8)	C2—N2—Ni2	129.31 (13)
N2 ⁱⁱ —Ni2—N2	90.88 (6)	N1—N2—Ni2	122.87 (12)
N2 ⁱ —Ni2—N2	90.88 (6)	C2—N3—C1	106.38 (16)
F2—Si1—F2 ^{vi}	91.80 (9)	C2—N3—H3	122 (2)
F2—Si1—F2 ^{vii}	91.80 (9)	C1—N3—H3	132 (2)
F2 ^{vi} —Si1—F2 ^{vii}	91.80 (9)	C2—N4—H4A	125 (2)
F2—Si1—F1 ^{vii}	90.36 (8)	C2—N4—H4B	122 (3)
F2 ^{vi} —Si1—F1 ^{vii}	177.12 (10)	H4A—N4—H4B	111 (3)
F2 ^{vii} —Si1—F1 ^{vii}	90.03 (8)	C1—N5—H5A	117 (2)
F2—Si1—F1 ^{vi}	177.12 (10)	C1—N5—H5B	116 (2)
F2 ^{vi} —Si1—F1 ^{vi}	90.03 (8)	H5A—N5—H5B	126 (3)
F2 ^{vii} —Si1—F1 ^{vi}	90.36 (8)	N1—C1—N5	127.37 (19)
F1 ^{vii} —Si1—F1 ^{vi}	87.74 (9)	N1—C1—N3	109.86 (16)
F2—Si1—F1	90.03 (8)	N5—C1—N3	122.76 (18)
F2 ^{vi} —Si1—F1	90.36 (8)	N2—C2—N4	126.92 (19)
F2 ^{vii} —Si1—F1	177.12 (10)	N2—C2—N3	110.33 (17)
F1 ^{vii} —Si1—F1	87.74 (9)	N4—C2—N3	122.67 (19)
N1 ⁱⁱ —Ni1—N1—C1	-135.1 (2)	N2 ^{iv} —Ni2—N2—N1	146.76 (16)
N1 ⁱ —Ni1—N1—C1	131.1 (2)	N2 ^v —Ni2—N2—N1	-122.40 (15)

supplementary materials

O1 ⁱ —Ni1—N1—C1	-44.81 (19)	N2 ⁱⁱ —Ni2—N2—N1	-31.71 (12)
O1 ⁱⁱ —Ni1—N1—C1	43.72 (19)	N2 ⁱ —Ni2—N2—N1	59.18 (10)
N1 ⁱⁱ —Ni1—N1—N2	60.39 (10)	N2—N1—C1—N5	-179.0 (2)
N1 ⁱ —Ni1—N1—N2	-33.46 (12)	Ni1—N1—C1—N5	14.9 (3)
O1 ⁱ —Ni1—N1—N2	150.67 (13)	N2—N1—C1—N3	-0.1 (2)
O1 ⁱⁱ —Ni1—N1—N2	-120.79 (13)	Ni1—N1—C1—N3	-166.30 (14)
C1—N1—N2—C2	0.6 (2)	C2—N3—C1—N1	-0.4 (2)
Ni1—N1—N2—C2	168.37 (13)	C2—N3—C1—N5	178.5 (2)
C1—N1—N2—Ni2	168.09 (13)	N1—N2—C2—N4	176.0 (2)
Ni1—N1—N2—Ni2	-24.17 (18)	Ni2—N2—C2—N4	9.6 (3)
N2 ^{iv} —Ni2—N2—C2	-48.86 (15)	N1—N2—C2—N3	-0.9 (2)
N2 ^v —Ni2—N2—C2	41.98 (14)	Ni2—N2—C2—N3	-167.25 (14)
N2 ⁱⁱ —Ni2—N2—C2	132.67 (18)	C1—N3—C2—N2	0.8 (2)
N2 ⁱ —Ni2—N2—C2	-136.44 (18)	C1—N3—C2—N4	-176.2 (2)

Symmetry codes: (i) $-y+1, x-y, z$; (ii) $-x+y+1, -x+1, z$; (iii) $y+1/3, x-1/3, -z+1/6$; (iv) $x-y+1/3, -y+2/3, -z+1/6$; (v) $-x+4/3, -x+y+2/3, -z+1/6$; (vi) $-y+1, x-y-1, z$; (vii) $-x+y+2, -x+1, z$; (viii) $-x+2/3, -y-2/3, -z+1/3$; (ix) $x-y-1/3, x-2/3, -z+1/3$; (x) $y+2/3, -x+y+1/3, -z+1/3$; (xi) $-y, x-y-1, z$; (xii) $-x+y+1, -x, z$.

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

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H11 \cdots O2	0.86 (3)	1.88 (3)	2.724 (3)	168 (3)
O1—H12 \cdots O3	0.85 (3)	1.89 (3)	2.737 (2)	174 (3)
O2—H21 \cdots O4	0.84 (3)	2.07 (3)	2.896 (4)	168 (4)
O2—H22 \cdots O3 ^{xiii}	0.84 (3)	2.12 (3)	2.961 (3)	175 (4)
O3—H31 \cdots O4 ^{ix}	0.85 (3)	2.05 (3)	2.861 (3)	159 (3)
O3—H32 \cdots F1 ^{ix}	0.84 (3)	2.13 (3)	2.904 (2)	153 (3)
O4—H41 \cdots F2 ^{xiv}	0.85 (3)	2.23 (3)	2.950 (3)	143 (4)
O4—H42 \cdots F3 ^{xi}	0.84 (3)	2.16 (3)	2.973 (4)	162 (4)
O4—H41 \cdots O5	0.85 (3)	2.03 (3)	2.64 (2)	128 (4)
O5—H51 \cdots F1	0.85	1.92	2.76 (3)	167
N3—H3 \cdots F2 ⁱⁱⁱ	0.85 (3)	1.97 (3)	2.764 (2)	157 (3)
N4—H4b \cdots F1 ⁱⁱⁱ	0.84 (3)	2.14 (3)	2.974 (3)	170 (3)
N5—H5a \cdots O1 ⁱ	0.84 (3)	2.23 (3)	2.942 (3)	142 (3)
N5—H5b \cdots F3 ^{xv}	0.85 (3)	2.08 (3)	2.909 (3)	167 (3)

Symmetry codes: (xiii) $-x+5/3, -y+1/3, -z+1/3$; (ix) $x-y-1/3, x-2/3, -z+1/3$; (xiv) $y+1/3, x-4/3, -z+1/6$; (xi) $-y, x-y-1, z$; (iii) $y+1/3, x-1/3, -z+1/6$; (i) $-y+1, x-y, z$; (xv) $x, y+1, z$.

Fig. 1

