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(μ_3-Benzene-1,3,5-tricarboxylato)tris[aqua(*N*,*N*,*N*′,*N*″,*N*″-pentamethyldiethylenetriamine)nickel(II)] tris(perchlorate) 4.25-hydrate

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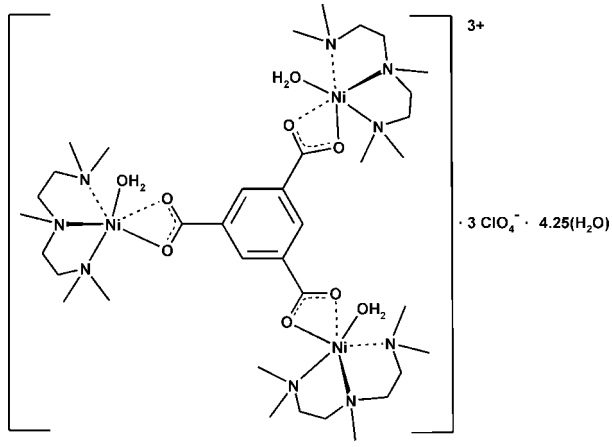
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.005 Å; disorder in solvent or counterion; R factor = 0.041; wR factor = 0.096; data-to-parameter ratio = 12.8.

In the title complex, $[Ni_3(C_9H_3O_6)(C_9H_{23}N_3)_3(H_2O)_3](ClO_4)_3\cdot 4.25H_2O$, the three Ni^{II} centres are bridged by a benzene-1,3,5-tricarboxylate(3−) anion (btc). Each of the metal centres is coordinated by three N atoms of a tridentate *N*,*N*,*N*′,*N*″,*N*″-pentamethyldiethylenetriamine ligand (pmdien), two O atoms of the btc ligand and one water molecule in a distorted octahedral geometry. The secondary structure is stabilized by a variety of O—H···O hydrogen bonds and C—H···O interactions that serve to connect the complex cations, perchlorate anions and water solvent molecules. Variability of intermolecular contacts stabilizing the crystal structure leads to disorder of part of O atoms of two perchlorate anions between two positions with occupancies 0.632 (5) and 0.337 (7).

Related literature

For related literature, see: Chen *et al.* (2005); Allen (2002); Daiguebonne *et al.* (1998, 2000, 2002); Kopel *et al.* (2007); Lin *et al.* (2005); Livage *et al.* (2001); Wang *et al.* (2004).



Experimental

Crystal data

$[Ni_3(C_9H_3O_6)(C_9H_{23}N_3)_3(H_2O)_3](ClO_4)_3\cdot 4.25H_2O$
M_r = 1332.12
Monoclinic, $P2_1/c$
a = 11.7409 (2) Å
b = 30.6727 (5) Å
c = 16.9590 (4) Å
β = 107.633 (2)°
V = 5820.41 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.18 mm^{-1}
T = 100 (2) K
0.30 × 0.30 × 0.25 mm

Data collection

Oxford Diffraction Xcalibur2 diffractometer with CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.655, T_{max} = 0.740
27090 measured reflections
10209 independent reflections
8075 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.096
S = 1.01
10209 reflections
799 parameters
26 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.01 e Å^{-3}
$\Delta\rho_{min}$ = −1.02 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3*V*···O52	0.915 (19)	1.77 (2)	2.677 (4)	173 (4)
O3—H3*W*···O7^{i}	0.93 (4)	1.83 (2)	2.742 (3)	167 (4)
O6—H6*W*···O51	0.91 (2)	1.99 (4)	2.607 (9)	124 (4)
O6—H6*V*···O54^{i}	0.930 (19)	1.83 (2)	2.745 (4)	166 (4)
O9—H9*V*···O1^{i}	0.908 (19)	1.85 (2)	2.743 (3)	166 (3)
O9—H9*W*···O55^{i}	0.913 (19)	1.82 (2)	2.727 (3)	171 (4)
O52—H52*V*···O53^{i}	0.95 (2)	2.13 (5)	2.789 (5)	125 (4)
O53—H53*W*···O7	0.99 (2)	1.97 (3)	2.877 (4)	151 (5)
O53—H53*V*···O97	1.00 (2)	1.96 (3)	2.869 (5)	151 (5)
O54—H54*V*···O2	0.933 (19)	1.98 (2)	2.902 (3)	167 (4)
O55—H55*V*···O4	0.914 (19)	1.95 (2)	2.843 (3)	166 (4)
O55—H55*W*···O76^{ii}	0.909 (19)	2.04 (2)	2.934 (4)	168 (4)

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97*

  doi:10.1107/S1600536807051938

(Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2201).

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supplementary materials

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(μ_3-Benzene-1,3,5-tricarboxylato)tris[aqua(*N*,*N*,*N*',*N*'',*N*''-pentamethyldiethylenetriamine)nickel(II)] tris(perchlorate) 4.25-hydrate

Z. Trávnícek, J. Marek and S. Cermáková

Comment

The molecular structure of the title complex, (I), is depicted in Fig. 1. The structure comprises a trinuclear Ni^{II} trication, three perchlorate anions and 4.25 crystal water molecules of crystallization. The three Ni^{II} metal centres are bridged by a benzene-1,3,5-tricarboxylate(3-) anion (btc). The atoms forming the Ni_3(btc) moiety deviate from planarity with dihedral angles between the central aromatic ring and the peripheral 4-membered C–O–Ni–O rings being in the range 3.2 (9)° to 8.1 (5)° for the rings containing Ni3 and Ni1 atoms, respectively (Brandenburg, 2006). Each of Ni^{II} cations is positioned at the vertex of an equilateral triangle with the Ni···Ni distances being 9.5197 (6) Å for Ni1···Ni2, 8.9876 (6) Å for Ni1···Ni3, and 9.6056 (5) Å for Ni2···Ni3. The geometry of the Ni centre is distorted octahedral. The degree of deformation can be seen from interatomic parameters around the Ni atom which differ significantly, *e.g.* Ni—O_{btc} bond lengths are in the range of 2.036 (2) to 2.493 (2) Å, whilst Ni—O(water) distances are similar and vary from 2.041 (2) to 2.079 (3) Å. For comparison, the Ni···Ni separations in the recently published polynuclear Ni complex involving the Ni_3(btc) moiety are 9.236 (4), 9.236 (6) and 9.294 (6) Å, and Ni—O bond lengths of a bidentate coordinated carboxylate group vary from 2.110 (8) to 2.130 (8) Å (Chen *et al.*, 2005).

To date, only 19 X-ray structures involving the M_3(btc) moiety, where *M* represents any transition metal, are included in the CSD (Cambridge Structural Database, Version 5.28.1; Allen, 2002), *e.g.* Gd (Daiguebonne *et al.*, 1998), Co (Livage *et al.*, 2001), In (Lin *et al.*, 2005), Nd (Wang *et al.*, 2004), Ni (Chen *et al.*, 2005), Sm (Daiguebonne *et al.*, 2002) and Er (Daiguebonne *et al.*, 2000).

The secondary structure of (I) is stabilized by a variety of O—H···O hydrogen bonds (Table 1, Fig. 2) and C—H···O interactions (Fig. 3), connecting the complex cations, perchlorate anions and water molecules.

Experimental

The title complex, (I), was prepared by a recently described method (Kopel *et al.*, 2007). Crystals suitable for X-ray analysis were obtained by recrystallization of (I) from water.

Refinement

Some atoms of perchlorate anions (namely O atoms O77, O78 and O79 bonded to Cl2, and O atoms O86, O88 and O89 bonded to Cl3) were refined as disordered between two positions [the site occupancy factors was refined to 0.632 (5) and 0.337 (7)]. C-bound H-atoms were included in the riding model approximation with C–H distances of 0.95 − 0.99 Å (CH_2), and with $U_{iso}(H) = 1.2U_{eq}(CH_2)$ or $1.5U_{eq}(C_{methyl})$. The O-bound H atoms were refined, with the O—H distances restrained to 0.95 (2) Å and with $U_{iso}(H)$ values of $1.5U_{eq}(O_{water})$; distances are given in Table 1. The maximum and minimum residual

electron denisty peaks were located in the neighbourhood of disordered perchlorate anions with distances 1.33 and 0.84 Å, respectively from the O88A and Cl2 atoms.

Figures

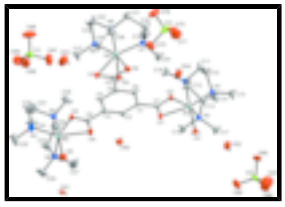


Fig. 1. The molecular structure of the title complex (I). The non-H atoms are drawn as 50% probability displacement ellipsoids. The disordered parts of the perchlorate anions have been omitted for clarity. The H-atoms are shown as short solid lines.

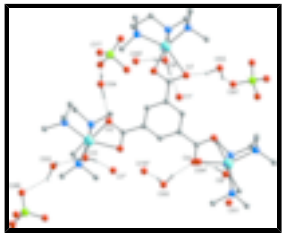


Fig. 2. Part of the crystal structure of (I), showing the formation of O—H···O hydrogen bonds [Symmetry codes: (i) −*x* + 1, −*y* + 1, −*z* + 1; (ii) −*x*, −*y* + 1, −*z* + 1]. Some of disordered perchlorate atoms and H-atoms not involved in hydrogen bonding are omitted for clarity.

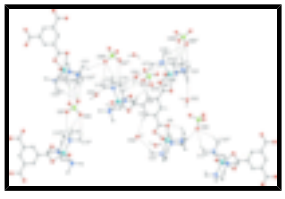


Fig. 3. Part of the crystal structure of (I), showing the formation of C—H···O contacts [Symmetry codes: (i) −*x* + 1, −*y* + 1, −*z* + 1; (ii) −*x*, −*y* + 1, −*z* + 1; (iii) 1 − *x*, −1/2 + *y*, 3/2 − *z*, (iv) *x*, 1/2 − *y*, −1/2 + *z*; (v) *x*, 1/2 − *y*, 1/2 + *z*; (vi) 1 − *x*, 1/2 + *y*, 3/2 − *z*; (vii) −*x*, 1/2 + *y*, 1/2 − *z*]. Some of disordered perchlorate atoms and H-atoms not involved in hydrogen bonding are omitted for clarity.

(μ$_3$-Benzene-1,3,5-tricarboxylato)tris[aqua(*N*,*N*,*N*',*N*'',*N*''- pentamethyldiethylenetriamine)nickel(II)] tris(perchlorate) 4.25-hydrate

Crystal data

$[Ni_3(C_9H_3O_6)(C_9H_{23}N_3)_3(H_2O)_3](ClO_4)_3·4.25H_2O$	F_{000} = 2810
M_r = 1332.12	D_x = 1.520 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 25991 reflections
a = 11.7409 (2) Å	θ = 2.6–32.0º
b = 30.6727 (5) Å	μ = 1.18 mm^{-1}
c = 16.9590 (4) Å	T = 100 (2) K
β = 107.633 (2)º	Prism, dark-green
V = 5820.41 (19) Å^3	0.30 × 0.30 × 0.25 mm
Z = 4	

Data collection

Oxford Diffraction Xcalibur2 + CCD diffractometer	10209 independent reflections
Radiation source: Enhance (Mo) X-ray Source	8075 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.025

Detector resolution: 8.3611 pixels mm^{-1}
T = 100(2) K
ω scans
Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2006)
T_{min} = 0.655, T_{max} = 0.740
27090 measured reflections

θ_{max} = 25.1º
θ_{min} = 2.6º
h = −13→13
k = −35→28
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.096
S = 1.01
10209 reflections
799 parameters
26 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.03P)^2 + 15P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 1.01 e Å^{-3}
$\Delta\rho_{min}$ = −1.02 e Å^{-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 $F^2 > \sigma(F^2)$ 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}	Occ. (<1)
Ni1	0.18931 (4)	0.372432 (13)	0.49777 (2)	0.01350 (10)	
Ni2	0.24189 (4)	0.612514 (14)	0.15475 (2)	0.01464 (10)	
Ni3	0.57687 (4)	0.611801 (13)	0.74650 (2)	0.01328 (10)	
O1	0.2105 (2)	0.42302 (7)	0.41337 (13)	0.0167 (5)	
O2	0.2956 (2)	0.42913 (7)	0.54701 (13)	0.0167 (5)	
O3	0.3311 (2)	0.34306 (8)	0.47357 (15)	0.0201 (5)	
H3V	0.371 (3)	0.3184 (9)	0.497 (2)	0.030*	
H3W	0.386 (3)	0.3588 (11)	0.455 (2)	0.030*	
O4	0.2287 (2)	0.55024 (7)	0.22527 (13)	0.0221 (5)	

O5	0.3051 (2)	0.61172 (7)	0.28366 (13)	0.0202 (5)
O6	0.4100 (2)	0.59180 (10)	0.15492 (16)	0.0314 (6)
H6V	0.460 (3)	0.5804 (14)	0.2039 (18)	0.047*
H6W	0.462 (3)	0.6017 (14)	0.128 (3)	0.047*
O7	0.5043 (2)	0.62186 (7)	0.59350 (14)	0.0201 (5)
O8	0.4838 (2)	0.56524 (8)	0.66740 (13)	0.0208 (5)
O9	0.7360 (2)	0.59170 (8)	0.73098 (14)	0.0179 (5)
H9V	0.741 (3)	0.5875 (12)	0.6791 (14)	0.027*
H9W	0.789 (3)	0.5725 (10)	0.764 (2)	0.027*
N1	0.2254 (3)	0.34506 (9)	0.61657 (17)	0.0187 (6)
N2	0.0337 (2)	0.39932 (9)	0.51974 (16)	0.0165 (6)
N3	0.0646 (2)	0.33156 (9)	0.41396 (16)	0.0163 (6)
N4	0.2765 (3)	0.68067 (10)	0.15369 (19)	0.0262 (7)
N5	0.0659 (2)	0.63192 (9)	0.14337 (17)	0.0201 (6)
N6	0.1648 (3)	0.59061 (10)	0.02995 (17)	0.0217 (6)
N7	0.5875 (2)	0.57154 (9)	0.84912 (16)	0.0184 (6)
N8	0.4263 (2)	0.63958 (9)	0.77056 (16)	0.0184 (6)
N9	0.6533 (2)	0.67424 (9)	0.78328 (16)	0.0168 (6)
C1	0.3081 (3)	0.49073 (10)	0.46320 (19)	0.0135 (7)
C2	0.2790 (3)	0.50910 (11)	0.38461 (19)	0.0155 (7)
H2	0.2363	0.4924	0.3378	0.019*
C3	0.3123 (3)	0.55197 (11)	0.37416 (19)	0.0158 (7)
C4	0.3748 (3)	0.57623 (10)	0.44331 (19)	0.0148 (7)
H4	0.3982	0.6053	0.4365	0.018*
C5	0.4032 (3)	0.55825 (10)	0.52214 (19)	0.0151 (7)
C6	0.3696 (3)	0.51542 (10)	0.53177 (19)	0.0151 (7)
H6	0.3889	0.5030	0.5855	0.018*
C7	0.2709 (3)	0.44529 (10)	0.47542 (19)	0.0142 (7)
C8	0.2798 (3)	0.57222 (11)	0.2895 (2)	0.0181 (7)
C9	0.4679 (3)	0.58317 (11)	0.59844 (19)	0.0163 (7)
C10	0.1662 (3)	0.37705 (12)	0.6569 (2)	0.0225 (8)
H10A	0.2098	0.4051	0.6643	0.027*
H10B	0.1679	0.3662	0.7122	0.027*
C11	0.0378 (3)	0.38396 (12)	0.6041 (2)	0.0218 (8)
H11A	−0.0070	0.3563	0.5996	0.026*
H11B	−0.0008	0.4058	0.6306	0.026*
C12	−0.0713 (3)	0.38080 (11)	0.4570 (2)	0.0229 (8)
H12A	−0.0898	0.3985	0.4058	0.027*
H12B	−0.1415	0.3818	0.4778	0.027*
C13	−0.0470 (3)	0.33401 (11)	0.4378 (2)	0.0202 (7)
H13A	−0.0391	0.3156	0.4871	0.024*
H13B	−0.1149	0.3229	0.3921	0.024*
C14	0.1844 (3)	0.30049 (12)	0.6262 (2)	0.0266 (8)
H14A	0.2016	0.2938	0.6852	0.040*
H14B	0.2262	0.2796	0.6010	0.040*
H14C	0.0981	0.2984	0.5989	0.040*
C15	0.3560 (3)	0.34564 (13)	0.6598 (2)	0.0260 (8)
H15A	0.3897	0.3734	0.6489	0.039*
H15B	0.3944	0.3216	0.6394	0.039*

H15C	0.3700	0.3423	0.7194	0.039*
C16	0.0250 (3)	0.44741 (11)	0.5169 (2)	0.0251 (8)
H16A	0.0267	0.4578	0.4627	0.038*
H16B	0.0926	0.4599	0.5601	0.038*
H16C	−0.0500	0.4564	0.5262	0.038*
C17	0.0983 (3)	0.28511 (11)	0.4114 (2)	0.0228 (8)
H17A	0.1048	0.2713	0.4647	0.034*
H17B	0.1754	0.2832	0.4003	0.034*
H17C	0.0371	0.2701	0.3674	0.034*
C18	0.0431 (3)	0.34967 (11)	0.3294 (2)	0.0204 (7)
H18A	−0.0186	0.3324	0.2898	0.031*
H18B	0.1173	0.3485	0.3144	0.031*
H18C	0.0165	0.3800	0.3283	0.031*
C20	0.1797 (4)	0.69925 (13)	0.1802 (3)	0.0408 (11)
H20A	0.1777	0.7312	0.1720	0.049*
H20B	0.1947	0.6935	0.2399	0.049*
C21	0.0620 (4)	0.68036 (13)	0.1327 (3)	0.0348 (10)
H21A	0.0439	0.6877	0.0733	0.042*
H21B	−0.0018	0.6928	0.1529	0.042*
C22	−0.0135 (3)	0.61055 (14)	0.0689 (2)	0.0284 (9)
H22A	−0.0902	0.6266	0.0501	0.034*
H22B	−0.0306	0.5804	0.0828	0.034*
C23	0.0423 (3)	0.60938 (13)	0.0003 (2)	0.0275 (8)
H23A	−0.0077	0.5915	−0.0459	0.033*
H23B	0.0462	0.6393	−0.0205	0.033*
C24	0.3929 (4)	0.69117 (15)	0.2159 (3)	0.0495 (13)
H24A	0.4008	0.7229	0.2228	0.074*
H24B	0.4582	0.6799	0.1969	0.074*
H24C	0.3964	0.6776	0.2689	0.074*
C25	0.2803 (4)	0.70070 (15)	0.0757 (3)	0.0484 (12)
H25A	0.2038	0.6959	0.0329	0.073*
H25B	0.3447	0.6874	0.0583	0.073*
H25C	0.2949	0.7321	0.0839	0.073*
C26	0.0262 (4)	0.62113 (16)	0.2159 (3)	0.0404 (11)
H26A	−0.0572	0.6301	0.2052	0.061*
H26B	0.0765	0.6365	0.2647	0.061*
H26C	0.0329	0.5896	0.2257	0.061*
C27	0.1576 (5)	0.54266 (14)	0.0316 (2)	0.0452 (12)
H27A	0.1102	0.5339	0.0676	0.068*
H27B	0.2383	0.5305	0.0529	0.068*
H27C	0.1196	0.5318	−0.0245	0.068*
C28	0.2286 (4)	0.60271 (17)	−0.0305 (2)	0.0434 (12)
H28A	0.3105	0.5915	−0.0114	0.065*
H28B	0.2304	0.6345	−0.0352	0.065*
H28C	0.1868	0.5901	−0.0846	0.065*
C30	0.4667 (3)	0.57526 (13)	0.8598 (2)	0.0298 (9)
H30A	0.4687	0.5637	0.9147	0.036*
H30B	0.4090	0.5577	0.8170	0.036*
C31	0.4271 (3)	0.62195 (13)	0.8527 (2)	0.0299 (9)

H31A	0.3460	0.6240	0.8587	0.036*	
H31B	0.4822	0.6393	0.8974	0.036*	
C32	0.4437 (3)	0.68742 (12)	0.7751 (2)	0.0258 (8)	
H32A	0.3910	0.7006	0.8044	0.031*	
H32B	0.4214	0.6997	0.7185	0.031*	
C33	0.5721 (3)	0.69852 (12)	0.8200 (2)	0.0248 (8)	
H33A	0.5847	0.7303	0.8161	0.030*	
H33B	0.5904	0.6908	0.8793	0.030*	
C34	0.6044 (4)	0.52518 (12)	0.8308 (2)	0.0280 (9)	
H34A	0.6860	0.5208	0.8285	0.042*	
H34B	0.5473	0.5173	0.7774	0.042*	
H34C	0.5910	0.5068	0.8744	0.042*	
C35	0.6812 (3)	0.58291 (12)	0.9259 (2)	0.0274 (8)	
H35A	0.6694	0.6130	0.9415	0.041*	
H35B	0.7597	0.5803	0.9172	0.041*	
H35C	0.6770	0.5631	0.9702	0.041*	
C36	0.3111 (3)	0.62924 (13)	0.7090 (2)	0.0284 (9)	
H36A	0.2462	0.6427	0.7257	0.043*	
H36B	0.2999	0.5976	0.7056	0.043*	
H36C	0.3103	0.6406	0.6549	0.043*	
C37	0.6541 (3)	0.69661 (11)	0.7058 (2)	0.0222 (8)	
H37A	0.5724	0.6982	0.6680	0.033*	
H37B	0.7045	0.6803	0.6795	0.033*	
H37C	0.6860	0.7262	0.7187	0.033*	
C38	0.7766 (3)	0.67569 (12)	0.8407 (2)	0.0217 (8)	
H38A	0.8290	0.6578	0.8185	0.033*	
H38B	0.7772	0.6644	0.8948	0.033*	
H38C	0.8053	0.7059	0.8467	0.033*	
Cl1	0.10417 (9)	0.75444 (3)	0.36681 (5)	0.0291 (2)	
O96	0.0428 (3)	0.77425 (12)	0.4183 (2)	0.0558 (10)	
O97	0.1431 (3)	0.71176 (11)	0.4000 (2)	0.0516 (9)	
O98	0.0216 (3)	0.74819 (11)	0.28622 (17)	0.0551 (9)	
O99	0.2026 (3)	0.78013 (11)	0.3648 (2)	0.0599 (10)	
Cl2	0.15471 (10)	0.50855 (3)	0.74728 (8)	0.0448 (3)	
O76	0.1141 (3)	0.54718 (10)	0.7734 (2)	0.0553 (9)	
O77	0.2529 (6)	0.4881 (2)	0.7936 (4)	0.0741 (18)	0.632 (5)
O78	0.1822 (5)	0.52275 (18)	0.6676 (3)	0.0445 (12)	0.632 (5)
O79	0.0522 (10)	0.4811 (3)	0.7122 (4)	0.056 (3)	0.632 (5)
O77A	0.2363 (10)	0.4990 (4)	0.8379 (6)	0.0741 (18)	0.368 (5)
O78A	0.2408 (8)	0.5090 (3)	0.7093 (6)	0.0445 (12)	0.368 (5)
O79A	0.0760 (19)	0.4731 (6)	0.7436 (9)	0.056 (3)	0.368 (5)
O87	0.2731 (3)	0.13286 (13)	0.3765 (3)	0.0711 (11)	
Cl3	0.3567 (2)	0.16715 (9)	0.40985 (12)	0.0277 (6)	0.663 (7)
O86	0.3186 (6)	0.18374 (16)	0.4762 (3)	0.0439 (15)	0.663 (7)
O88	0.4662 (4)	0.14462 (16)	0.4400 (3)	0.0447 (15)	0.663 (7)
O89	0.3589 (7)	0.1989 (2)	0.3509 (4)	0.065 (2)	0.663 (7)
Cl3A	0.3081 (7)	0.17759 (15)	0.4139 (3)	0.0338 (13)	0.337 (7)
O86A	0.2359 (11)	0.1898 (4)	0.4636 (6)	0.048 (3)	0.337 (7)
O88A	0.4306 (10)	0.1809 (4)	0.4639 (7)	0.065 (4)	0.337 (7)

O89A	0.2786 (15)	0.2004 (6)	0.3427 (7)	0.082 (5)	0.337 (7)
O51	0.5580 (8)	0.5631 (3)	0.0792 (5)	0.0141 (18)*	0.25
H51W	0.603 (10)	0.589 (2)	0.092 (8)	0.021*	0.25
H51V	0.600 (10)	0.537 (2)	0.099 (8)	0.021*	0.25
O52	0.4498 (3)	0.26949 (10)	0.5315 (2)	0.0464 (8)	
H52V	0.532 (2)	0.2783 (17)	0.551 (3)	0.070*	
H52W	0.437 (5)	0.2448 (12)	0.497 (3)	0.070*	
O53	0.3673 (3)	0.69819 (11)	0.5254 (2)	0.0527 (9)	
H53V	0.301 (4)	0.6950 (18)	0.473 (2)	0.079*	
H53W	0.390 (5)	0.6671 (8)	0.536 (3)	0.079*	
O54	0.4280 (2)	0.45053 (9)	0.71672 (17)	0.0337 (7)	
H54V	0.396 (4)	0.4442 (15)	0.6605 (14)	0.051*	
H54W	0.372 (3)	0.4644 (14)	0.737 (3)	0.051*	
O55	0.1060 (2)	0.46955 (8)	0.18374 (15)	0.0240 (5)	
H55V	0.137 (3)	0.4963 (8)	0.202 (2)	0.036*	
H55W	0.038 (2)	0.4686 (14)	0.199 (2)	0.036*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ni1	0.0180 (2)	0.0101 (2)	0.0119 (2)	−0.00242 (17)	0.00381 (17)	0.00048 (16)
Ni2	0.0188 (2)	0.0142 (2)	0.0104 (2)	0.00220 (17)	0.00359 (17)	0.00279 (16)
Ni3	0.0161 (2)	0.0127 (2)	0.0099 (2)	−0.00199 (17)	0.00225 (16)	−0.00155 (16)
O1	0.0238 (12)	0.0128 (12)	0.0123 (11)	−0.0053 (10)	0.0037 (10)	−0.0014 (9)
O2	0.0246 (13)	0.0132 (12)	0.0112 (11)	−0.0036 (10)	0.0039 (10)	0.0011 (9)
O3	0.0223 (13)	0.0155 (13)	0.0257 (13)	0.0018 (10)	0.0122 (11)	0.0044 (10)
O4	0.0358 (14)	0.0153 (12)	0.0109 (11)	0.0004 (11)	0.0005 (11)	0.0007 (10)
O5	0.0328 (14)	0.0124 (12)	0.0131 (11)	0.0000 (10)	0.0034 (10)	0.0019 (9)
O6	0.0211 (14)	0.0443 (17)	0.0267 (14)	0.0087 (12)	0.0041 (11)	0.0030 (13)
O7	0.0254 (13)	0.0146 (13)	0.0226 (12)	−0.0069 (10)	0.0105 (11)	−0.0053 (10)
O8	0.0296 (14)	0.0193 (13)	0.0104 (11)	−0.0080 (10)	0.0016 (10)	−0.0031 (9)
O9	0.0208 (13)	0.0183 (13)	0.0154 (12)	0.0037 (10)	0.0068 (10)	−0.0008 (10)
N1	0.0251 (16)	0.0163 (15)	0.0151 (14)	0.0008 (12)	0.0070 (12)	0.0020 (11)
N2	0.0189 (15)	0.0126 (15)	0.0167 (14)	0.0003 (11)	0.0034 (12)	0.0011 (11)
N3	0.0191 (14)	0.0117 (14)	0.0170 (14)	−0.0033 (11)	0.0038 (12)	−0.0007 (11)
N4	0.0261 (17)	0.0185 (16)	0.0258 (16)	−0.0022 (13)	−0.0046 (13)	0.0096 (13)
N5	0.0206 (15)	0.0230 (16)	0.0175 (14)	0.0028 (12)	0.0069 (12)	0.0005 (12)
N6	0.0240 (16)	0.0279 (17)	0.0138 (14)	0.0045 (13)	0.0065 (12)	0.0012 (12)
N7	0.0230 (15)	0.0180 (15)	0.0127 (14)	0.0021 (12)	0.0032 (12)	0.0010 (11)
N8	0.0165 (14)	0.0236 (16)	0.0145 (14)	0.0029 (12)	0.0039 (12)	0.0032 (12)
N9	0.0191 (14)	0.0140 (15)	0.0161 (14)	0.0001 (11)	0.0035 (12)	−0.0034 (11)
C1	0.0143 (16)	0.0117 (16)	0.0143 (16)	−0.0014 (13)	0.0042 (13)	−0.0005 (13)
C2	0.0213 (17)	0.0146 (17)	0.0101 (15)	0.0013 (14)	0.0038 (13)	−0.0022 (13)
C3	0.0219 (18)	0.0137 (17)	0.0116 (16)	0.0030 (14)	0.0046 (14)	0.0016 (13)
C4	0.0216 (17)	0.0084 (16)	0.0158 (16)	−0.0014 (13)	0.0076 (14)	−0.0009 (13)
C5	0.0183 (17)	0.0146 (17)	0.0125 (16)	−0.0009 (13)	0.0046 (13)	0.0000 (13)
C6	0.0203 (17)	0.0149 (17)	0.0106 (15)	−0.0016 (13)	0.0053 (14)	0.0005 (13)
C7	0.0154 (16)	0.0132 (17)	0.0145 (16)	−0.0009 (13)	0.0052 (13)	−0.0024 (13)

C8	0.0236 (18)	0.0140 (18)	0.0163 (17)	0.0028 (14)	0.0057 (14)	−0.0007 (14)
C9	0.0191 (17)	0.0152 (18)	0.0161 (17)	−0.0017 (14)	0.0073 (14)	−0.0041 (14)
C10	0.030 (2)	0.025 (2)	0.0146 (17)	−0.0001 (16)	0.0100 (15)	0.0005 (14)
C11	0.0272 (19)	0.0227 (19)	0.0183 (17)	−0.0015 (15)	0.0109 (15)	0.0001 (14)
C12	0.0186 (18)	0.024 (2)	0.0247 (19)	−0.0020 (15)	0.0049 (15)	−0.0004 (15)
C13	0.0163 (17)	0.0210 (19)	0.0219 (18)	−0.0054 (14)	0.0035 (14)	0.0005 (14)
C14	0.034 (2)	0.0173 (19)	0.027 (2)	0.0006 (16)	0.0073 (17)	0.0093 (15)
C15	0.027 (2)	0.029 (2)	0.0185 (18)	0.0030 (16)	0.0013 (15)	0.0048 (15)
C16	0.032 (2)	0.0138 (19)	0.031 (2)	0.0033 (15)	0.0111 (17)	0.0008 (15)
C17	0.0269 (19)	0.0135 (18)	0.0262 (19)	−0.0031 (15)	0.0052 (16)	−0.0031 (15)
C18	0.0249 (19)	0.0186 (19)	0.0161 (17)	−0.0031 (15)	0.0040 (15)	−0.0021 (14)
C20	0.050 (3)	0.017 (2)	0.051 (3)	0.0067 (19)	0.010 (2)	−0.0016 (19)
C21	0.030 (2)	0.027 (2)	0.040 (2)	0.0135 (17)	0.0000 (19)	−0.0060 (18)
C22	0.0180 (18)	0.038 (2)	0.027 (2)	−0.0028 (17)	0.0040 (16)	−0.0086 (17)
C23	0.0215 (19)	0.036 (2)	0.0207 (18)	0.0013 (17)	0.0002 (15)	−0.0027 (16)
C24	0.048 (3)	0.030 (2)	0.049 (3)	−0.013 (2)	−0.017 (2)	0.012 (2)
C25	0.051 (3)	0.042 (3)	0.047 (3)	−0.011 (2)	0.007 (2)	0.023 (2)
C26	0.033 (2)	0.065 (3)	0.030 (2)	0.002 (2)	0.0186 (19)	0.003 (2)
C27	0.073 (3)	0.031 (2)	0.021 (2)	0.009 (2)	−0.001 (2)	−0.0075 (18)
C28	0.040 (2)	0.075 (3)	0.018 (2)	−0.002 (2)	0.0127 (18)	−0.001 (2)
C30	0.027 (2)	0.040 (2)	0.025 (2)	0.0009 (17)	0.0116 (17)	0.0117 (17)
C31	0.028 (2)	0.044 (3)	0.0229 (19)	0.0101 (18)	0.0154 (16)	0.0111 (17)
C32	0.028 (2)	0.023 (2)	0.027 (2)	0.0094 (16)	0.0104 (17)	−0.0009 (16)
C33	0.033 (2)	0.0173 (19)	0.0248 (19)	0.0051 (16)	0.0089 (17)	−0.0048 (15)
C34	0.041 (2)	0.0175 (19)	0.0221 (19)	−0.0022 (17)	0.0050 (17)	0.0033 (15)
C35	0.035 (2)	0.027 (2)	0.0151 (17)	−0.0016 (17)	−0.0003 (16)	−0.0011 (15)
C36	0.0153 (18)	0.036 (2)	0.030 (2)	−0.0010 (16)	0.0017 (16)	0.0052 (17)
C37	0.029 (2)	0.0155 (18)	0.0209 (18)	−0.0055 (15)	0.0062 (16)	−0.0012 (14)
C38	0.0187 (18)	0.0194 (19)	0.0239 (18)	−0.0036 (14)	0.0018 (15)	−0.0054 (15)
Cl1	0.0371 (5)	0.0292 (5)	0.0210 (4)	−0.0014 (4)	0.0090 (4)	−0.0033 (4)
O96	0.0341 (17)	0.080 (3)	0.053 (2)	−0.0077 (17)	0.0141 (15)	−0.0404 (19)
O97	0.0434 (19)	0.046 (2)	0.060 (2)	0.0035 (15)	0.0082 (16)	0.0241 (16)
O98	0.081 (2)	0.056 (2)	0.0203 (15)	−0.0005 (18)	0.0030 (16)	−0.0032 (14)
O99	0.060 (2)	0.050 (2)	0.085 (3)	−0.0138 (17)	0.043 (2)	−0.0031 (19)
Cl2	0.0406 (6)	0.0256 (6)	0.0790 (8)	−0.0072 (5)	0.0345 (6)	−0.0163 (5)
O76	0.054 (2)	0.0355 (19)	0.085 (3)	−0.0025 (15)	0.0348 (19)	−0.0224 (17)
O77	0.072 (4)	0.082 (4)	0.077 (5)	0.038 (3)	0.035 (3)	0.019 (4)
O78	0.049 (3)	0.052 (3)	0.040 (3)	−0.008 (2)	0.023 (2)	0.002 (2)
O79	0.081 (5)	0.046 (4)	0.057 (6)	−0.043 (4)	0.047 (5)	−0.025 (4)
O77A	0.072 (4)	0.082 (4)	0.077 (5)	0.038 (3)	0.035 (3)	0.019 (4)
O78A	0.049 (3)	0.052 (3)	0.040 (3)	−0.008 (2)	0.023 (2)	0.002 (2)
O79A	0.081 (5)	0.046 (4)	0.057 (6)	−0.043 (4)	0.047 (5)	−0.025 (4)
O87	0.057 (2)	0.070 (3)	0.079 (3)	−0.018 (2)	0.011 (2)	−0.024 (2)
Cl3	0.0230 (11)	0.0480 (13)	0.0128 (8)	0.0034 (9)	0.0065 (8)	0.0075 (8)
O86	0.071 (5)	0.048 (3)	0.021 (3)	0.021 (3)	0.025 (3)	0.005 (2)
O88	0.044 (3)	0.055 (3)	0.038 (3)	0.015 (2)	0.017 (2)	0.008 (2)
O89	0.095 (5)	0.060 (4)	0.057 (4)	0.017 (4)	0.048 (4)	0.028 (3)
Cl3A	0.047 (3)	0.029 (2)	0.023 (2)	0.000 (2)	0.008 (2)	0.0031 (14)
O86A	0.044 (7)	0.058 (7)	0.037 (6)	0.005 (5)	0.007 (5)	0.001 (5)

O88A	0.053 (7)	0.079 (9)	0.070 (8)	0.000 (6)	0.030 (7)	−0.029 (6)
O89A	0.086 (10)	0.142 (13)	0.018 (5)	0.006 (10)	0.015 (7)	0.039 (6)
O52	0.066 (2)	0.0275 (17)	0.050 (2)	0.0073 (16)	0.0249 (18)	0.0089 (14)
O53	0.053 (2)	0.0406 (19)	0.055 (2)	0.0109 (16)	0.0022 (17)	−0.0007 (16)
O54	0.0326 (16)	0.0237 (15)	0.0336 (16)	0.0035 (12)	−0.0068 (13)	−0.0040 (12)
O55	0.0247 (14)	0.0230 (14)	0.0229 (13)	0.0022 (11)	0.0053 (11)	−0.0030 (11)

Geometric parameters (Å, °)

Ni1—O3	2.041 (2)	C16—H16B	0.9800
Ni1—N1	2.104 (3)	C16—H16C	0.9800
Ni1—N3	2.115 (3)	C17—H17A	0.9800
Ni1—N2	2.138 (3)	C17—H17B	0.9800
Ni1—O2	2.156 (2)	C17—H17C	0.9800
Ni1—O1	2.176 (2)	C18—H18A	0.9800
Ni1—C7	2.505 (3)	C18—H18B	0.9800
Ni2—O6	2.072 (3)	C18—H18C	0.9800
Ni2—O5	2.085 (2)	C20—C21	1.490 (6)
Ni2—N5	2.102 (3)	C20—H20A	0.9900
Ni2—N4	2.131 (3)	C20—H20B	0.9900
Ni2—N6	2.142 (3)	C21—H21A	0.9900
Ni2—O4	2.284 (2)	C21—H21B	0.9900
Ni2—C8	2.517 (3)	C22—C23	1.497 (5)
Ni3—O8	2.036 (2)	C22—H22A	0.9900
Ni3—O9	2.059 (2)	C22—H22B	0.9900
Ni3—N7	2.107 (3)	C23—H23A	0.9900
Ni3—N8	2.111 (3)	C23—H23B	0.9900
Ni3—N9	2.127 (3)	C24—H24A	0.9800
O1—C7	1.275 (4)	C24—H24B	0.9800
O2—C7	1.261 (4)	C24—H24C	0.9800
O3—H3V	0.915 (19)	C25—H25A	0.9800
O3—H3W	0.93 (4)	C25—H25B	0.9800
O4—C8	1.267 (4)	C25—H25C	0.9800
O5—C8	1.259 (4)	C26—H26A	0.9800
O6—H6V	0.930 (19)	C26—H26B	0.9800
O6—H6W	0.91 (2)	C26—H26C	0.9800
O7—C9	1.273 (4)	C27—H27A	0.9800
O8—C9	1.254 (4)	C27—H27B	0.9800
O9—H9V	0.908 (19)	C27—H27C	0.9800
O9—H9W	0.913 (19)	C28—H28A	0.9800
N1—C14	1.475 (4)	C28—H28B	0.9800
N1—C10	1.484 (4)	C28—H28C	0.9800
N1—C15	1.485 (4)	C30—C31	1.499 (5)
N2—C12	1.477 (4)	C30—H30A	0.9900
N2—C16	1.479 (4)	C30—H30B	0.9900
N2—C11	1.493 (4)	C31—H31A	0.9900
N3—C17	1.483 (4)	C31—H31B	0.9900
N3—C13	1.486 (4)	C32—C33	1.508 (5)
N3—C18	1.486 (4)	C32—H32A	0.9900

N4—C20	1.459 (5)	C32—H32B	0.9900
N4—C25	1.471 (5)	C33—H33A	0.9900
N4—C24	1.487 (5)	C33—H33B	0.9900
N5—C22	1.477 (4)	C34—H34A	0.9800
N5—C26	1.479 (5)	C34—H34B	0.9800
N5—C21	1.496 (5)	C34—H34C	0.9800
N6—C27	1.474 (5)	C35—H35A	0.9800
N6—C28	1.488 (5)	C35—H35B	0.9800
N6—C23	1.488 (4)	C35—H35C	0.9800
N7—C35	1.470 (4)	C36—H36A	0.9800
N7—C34	1.481 (4)	C36—H36B	0.9800
N7—C30	1.487 (5)	C36—H36C	0.9800
N8—C36	1.471 (4)	C37—H37A	0.9800
N8—C32	1.480 (5)	C37—H37B	0.9800
N8—C31	1.491 (4)	C37—H37C	0.9800
N9—C38	1.481 (4)	C38—H38A	0.9800
N9—C37	1.485 (4)	C38—H38B	0.9800
N9—C33	1.487 (4)	C38—H38C	0.9800
C1—C2	1.391 (4)	Cl1—O99	1.408 (3)
C1—C6	1.392 (4)	Cl1—O96	1.426 (3)
C1—C7	1.494 (4)	Cl1—O98	1.429 (3)
C2—C3	1.398 (5)	Cl1—O97	1.444 (3)
C2—H2	0.9500	Cl2—O77	1.338 (6)
C3—C4	1.395 (4)	Cl2—O78A	1.353 (8)
C3—C8	1.503 (4)	Cl2—O76	1.398 (3)
C4—C5	1.390 (4)	Cl2—O79A	1.416 (12)
C4—H4	0.9500	Cl2—O79	1.440 (7)
C5—C6	1.395 (4)	Cl2—O78	1.544 (5)
C5—C9	1.497 (4)	Cl2—O77A	1.573 (10)
C6—H6	0.9500	O87—Cl3	1.433 (4)
C10—C11	1.518 (5)	O87—Cl3A	1.515 (6)
C10—H10A	0.9900	Cl3—O89	1.401 (6)
C10—H10B	0.9900	Cl3—O88	1.411 (5)
C11—H11A	0.9900	Cl3—O86	1.425 (5)
C11—H11B	0.9900	Cl3A—O89A	1.348 (12)
C12—C13	1.517 (5)	Cl3A—O86A	1.414 (12)
C12—H12A	0.9900	Cl3A—O88A	1.435 (13)
C12—H12B	0.9900	O51—H51W	0.94 (2)
C13—H13A	0.9900	O51—H51V	0.94 (2)
C13—H13B	0.9900	O52—H52V	0.95 (2)
C14—H14A	0.9800	O52—H52W	0.95 (2)
C14—H14B	0.9800	O53—H53V	1.00 (2)
C14—H14C	0.9800	O53—H53W	0.99 (2)
C15—H15A	0.9800	O54—H54V	0.933 (19)
C15—H15B	0.9800	O54—H54W	0.92 (2)
C15—H15C	0.9800	O55—H55V	0.914 (19)
C16—H16A	0.9800	O55—H55W	0.909 (19)
O3—Ni1—N1	94.38 (10)	N1—C14—H14B	109.5
O3—Ni1—N3	92.53 (10)	H14A—C14—H14B	109.5

N1—Ni1—N3	107.73 (11)	N1—C14—H14C	109.5
O3—Ni1—N2	176.07 (10)	H14A—C14—H14C	109.5
N1—Ni1—N2	85.52 (11)	H14B—C14—H14C	109.5
N3—Ni1—N2	83.77 (10)	N1—C15—H15A	109.5
O3—Ni1—O2	91.26 (9)	N1—C15—H15B	109.5
N1—Ni1—O2	91.48 (10)	H15A—C15—H15B	109.5
N3—Ni1—O2	160.05 (9)	N1—C15—H15C	109.5
N2—Ni1—O2	92.66 (9)	H15A—C15—H15C	109.5
O3—Ni1—O1	85.40 (9)	H15B—C15—H15C	109.5
N1—Ni1—O1	152.20 (10)	N2—C16—H16A	109.5
N3—Ni1—O1	100.04 (9)	N2—C16—H16B	109.5
N2—Ni1—O1	96.52 (9)	H16A—C16—H16B	109.5
O2—Ni1—O1	60.76 (8)	N2—C16—H16C	109.5
O3—Ni1—C7	89.48 (10)	H16A—C16—H16C	109.5
N1—Ni1—C7	121.70 (11)	H16B—C16—H16C	109.5
N3—Ni1—C7	130.21 (10)	N3—C17—H17A	109.5
N2—Ni1—C7	93.91 (10)	N3—C17—H17B	109.5
O2—Ni1—C7	30.23 (9)	H17A—C17—H17B	109.5
O1—Ni1—C7	30.59 (9)	N3—C17—H17C	109.5
O6—Ni2—O5	87.62 (10)	H17A—C17—H17C	109.5
O6—Ni2—N5	174.88 (11)	H17B—C17—H17C	109.5
O5—Ni2—N5	97.32 (10)	N3—C18—H18A	109.5
O6—Ni2—N4	96.70 (12)	N3—C18—H18B	109.5
O5—Ni2—N4	90.69 (10)	H18A—C18—H18B	109.5
N5—Ni2—N4	84.62 (12)	N3—C18—H18C	109.5
O6—Ni2—N6	91.28 (11)	H18A—C18—H18C	109.5
O5—Ni2—N6	160.36 (10)	H18B—C18—H18C	109.5
N5—Ni2—N6	83.61 (11)	N4—C20—C21	111.3 (3)
N4—Ni2—N6	108.90 (11)	N4—C20—H20A	109.4
O6—Ni2—O4	87.62 (10)	C21—C20—H20A	109.4
O5—Ni2—O4	60.02 (8)	N4—C20—H20B	109.4
N5—Ni2—O4	93.68 (10)	C21—C20—H20B	109.4
N4—Ni2—O4	150.28 (10)	H20A—C20—H20B	108.0
N6—Ni2—O4	100.35 (10)	C20—C21—N5	109.3 (3)
O6—Ni2—C8	86.66 (11)	C20—C21—H21A	109.8
O5—Ni2—C8	29.91 (10)	N5—C21—H21A	109.8
N5—Ni2—C8	96.95 (11)	C20—C21—H21B	109.8
N4—Ni2—C8	120.52 (11)	N5—C21—H21B	109.8
N6—Ni2—C8	130.45 (11)	H21A—C21—H21B	108.3
O4—Ni2—C8	30.12 (9)	N5—C22—C23	111.3 (3)
O8—Ni3—O9	91.85 (10)	N5—C22—H22A	109.4
O8—Ni3—N7	91.07 (10)	C23—C22—H22A	109.4
O9—Ni3—N7	95.81 (10)	N5—C22—H22B	109.4
O8—Ni3—N8	95.56 (10)	C23—C22—H22B	109.4
O9—Ni3—N8	172.46 (10)	H22A—C22—H22B	108.0
N7—Ni3—N8	85.43 (11)	N6—C23—C22	110.6 (3)
O8—Ni3—N9	156.85 (10)	N6—C23—H23A	109.5
O9—Ni3—N9	89.35 (10)	C22—C23—H23A	109.5
N7—Ni3—N9	111.81 (11)	N6—C23—H23B	109.5

N8—Ni3—N9	83.30 (11)	C22—C23—H23B	109.5
C7—O1—Ni1	89.13 (18)	H23A—C23—H23B	108.1
C7—O2—Ni1	90.36 (18)	N4—C24—H24A	109.5
Ni1—O3—H3V	128 (2)	N4—C24—H24B	109.5
Ni1—O3—H3W	122 (2)	H24A—C24—H24B	109.5
H3V—O3—H3W	105 (3)	N4—C24—H24C	109.5
C8—O4—Ni2	85.16 (19)	H24A—C24—H24C	109.5
C8—O5—Ni2	94.38 (19)	H24B—C24—H24C	109.5
Ni2—O6—H6V	118 (3)	N4—C25—H25A	109.5
Ni2—O6—H6W	132 (3)	N4—C25—H25B	109.5
H6V—O6—H6W	103 (4)	H25A—C25—H25B	109.5
C9—O8—Ni3	101.9 (2)	N4—C25—H25C	109.5
Ni3—O9—H9V	119 (2)	H25A—C25—H25C	109.5
Ni3—O9—H9W	126 (2)	H25B—C25—H25C	109.5
H9V—O9—H9W	106 (3)	N5—C26—H26A	109.5
C14—N1—C10	110.3 (3)	N5—C26—H26B	109.5
C14—N1—C15	106.2 (3)	H26A—C26—H26B	109.5
C10—N1—C15	108.8 (3)	N5—C26—H26C	109.5
C14—N1—Ni1	119.9 (2)	H26A—C26—H26C	109.5
C10—N1—Ni1	101.01 (19)	H26B—C26—H26C	109.5
C15—N1—Ni1	110.2 (2)	N6—C27—H27A	109.5
C12—N2—C16	109.1 (3)	N6—C27—H27B	109.5
C12—N2—C11	110.3 (3)	H27A—C27—H27B	109.5
C16—N2—C11	109.1 (3)	N6—C27—H27C	109.5
C12—N2—Ni1	107.3 (2)	H27A—C27—H27C	109.5
C16—N2—Ni1	115.7 (2)	H27B—C27—H27C	109.5
C11—N2—Ni1	105.3 (2)	N6—C28—H28A	109.5
C17—N3—C13	108.8 (3)	N6—C28—H28B	109.5
C17—N3—C18	107.4 (3)	H28A—C28—H28B	109.5
C13—N3—C18	109.8 (3)	N6—C28—H28C	109.5
C17—N3—Ni1	116.9 (2)	H28A—C28—H28C	109.5
C13—N3—Ni1	105.96 (19)	H28B—C28—H28C	109.5
C18—N3—Ni1	107.90 (19)	N7—C30—C31	110.3 (3)
C20—N4—C25	110.6 (3)	N7—C30—H30A	109.6
C20—N4—C24	109.3 (3)	C31—C30—H30A	109.6
C25—N4—C24	106.0 (3)	N7—C30—H30B	109.6
C20—N4—Ni2	102.2 (2)	C31—C30—H30B	109.6
C25—N4—Ni2	118.4 (3)	H30A—C30—H30B	108.1
C24—N4—Ni2	110.1 (2)	N8—C31—C30	109.6 (3)
C22—N5—C26	109.6 (3)	N8—C31—H31A	109.7
C22—N5—C21	110.4 (3)	C30—C31—H31A	109.7
C26—N5—C21	108.5 (3)	N8—C31—H31B	109.7
C22—N5—Ni2	107.5 (2)	C30—C31—H31B	109.7
C26—N5—Ni2	114.2 (2)	H31A—C31—H31B	108.2
C21—N5—Ni2	106.6 (2)	N8—C32—C33	110.6 (3)
C27—N6—C28	107.8 (3)	N8—C32—H32A	109.5
C27—N6—C23	109.7 (3)	C33—C32—H32A	109.5
C28—N6—C23	107.9 (3)	N8—C32—H32B	109.5
C27—N6—Ni2	107.6 (2)	C33—C32—H32B	109.5

C28—N6—Ni2	116.9 (2)	H32A—C32—H32B	108.1
C23—N6—Ni2	106.7 (2)	N9—C33—C32	110.2 (3)
C35—N7—C34	107.6 (3)	N9—C33—H33A	109.6
C35—N7—C30	111.1 (3)	C32—C33—H33A	109.6
C34—N7—C30	107.0 (3)	N9—C33—H33B	109.6
C35—N7—Ni3	115.7 (2)	C32—C33—H33B	109.6
C34—N7—Ni3	111.6 (2)	H33A—C33—H33B	108.1
C30—N7—Ni3	103.6 (2)	N7—C34—H34A	109.5
C36—N8—C32	109.5 (3)	N7—C34—H34B	109.5
C36—N8—C31	109.2 (3)	H34A—C34—H34B	109.5
C32—N8—C31	110.4 (3)	N7—C34—H34C	109.5
C36—N8—Ni3	114.8 (2)	H34A—C34—H34C	109.5
C32—N8—Ni3	107.4 (2)	H34B—C34—H34C	109.5
C31—N8—Ni3	105.4 (2)	N7—C35—H35A	109.5
C38—N9—C37	107.5 (3)	N7—C35—H35B	109.5
C38—N9—C33	109.5 (3)	H35A—C35—H35B	109.5
C37—N9—C33	108.4 (3)	N7—C35—H35C	109.5
C38—N9—Ni3	117.5 (2)	H35A—C35—H35C	109.5
C37—N9—Ni3	105.77 (19)	H35B—C35—H35C	109.5
C33—N9—Ni3	107.9 (2)	N8—C36—H36A	109.5
C2—C1—C6	119.6 (3)	N8—C36—H36B	109.5
C2—C1—C7	120.9 (3)	H36A—C36—H36B	109.5
C6—C1—C7	119.4 (3)	N8—C36—H36C	109.5
C1—C2—C3	120.4 (3)	H36A—C36—H36C	109.5
C1—C2—H2	119.8	H36B—C36—H36C	109.5
C3—C2—H2	119.8	N9—C37—H37A	109.5
C4—C3—C2	119.4 (3)	N9—C37—H37B	109.5
C4—C3—C8	119.9 (3)	H37A—C37—H37B	109.5
C2—C3—C8	120.6 (3)	N9—C37—H37C	109.5
C5—C4—C3	120.6 (3)	H37A—C37—H37C	109.5
C5—C4—H4	119.7	H37B—C37—H37C	109.5
C3—C4—H4	119.7	N9—C38—H38A	109.5
C4—C5—C6	119.5 (3)	N9—C38—H38B	109.5
C4—C5—C9	122.7 (3)	H38A—C38—H38B	109.5
C6—C5—C9	117.8 (3)	N9—C38—H38C	109.5
C1—C6—C5	120.5 (3)	H38A—C38—H38C	109.5
C1—C6—H6	119.7	H38B—C38—H38C	109.5
C5—C6—H6	119.7	O99—Cl1—O96	110.4 (2)
O2—C7—O1	119.5 (3)	O99—Cl1—O98	112.1 (2)
O2—C7—C1	120.6 (3)	O96—Cl1—O98	108.7 (2)
O1—C7—C1	119.9 (3)	O99—Cl1—O97	110.6 (2)
O2—C7—Ni1	59.41 (16)	O96—Cl1—O97	107.9 (2)
O1—C7—Ni1	60.28 (16)	O98—Cl1—O97	107.1 (2)
C1—C7—Ni1	174.1 (2)	O77—Cl2—O78A	69.5 (5)
O5—C8—O4	120.4 (3)	O77—Cl2—O76	121.8 (4)
O5—C8—C3	118.4 (3)	O78A—Cl2—O76	121.1 (4)
O4—C8—C3	121.2 (3)	O77—Cl2—O79A	95.8 (10)
O5—C8—Ni2	55.70 (16)	O78A—Cl2—O79A	124.0 (10)
O4—C8—Ni2	64.72 (17)	O76—Cl2—O79A	112.6 (11)

C3—C8—Ni2	173.7 (2)	O77—Cl2—O79	115.7 (6)
O8—C9—O7	120.8 (3)	O78A—Cl2—O79	117.4 (6)
O8—C9—C5	118.4 (3)	O76—Cl2—O79	107.7 (6)
O7—C9—C5	120.8 (3)	O77—Cl2—O78	106.0 (4)
N1—C10—C11	109.9 (3)	O76—Cl2—O78	102.7 (3)
N1—C10—H10A	109.7	O79A—Cl2—O78	119.0 (7)
C11—C10—H10A	109.7	O79—Cl2—O78	99.9 (4)
N1—C10—H10B	109.7	O78A—Cl2—O77A	98.4 (6)
C11—C10—H10B	109.7	O76—Cl2—O77A	90.8 (5)
H10A—C10—H10B	108.2	O79A—Cl2—O77A	95.5 (8)
N2—C11—C10	110.3 (3)	O79—Cl2—O77A	118.7 (6)
N2—C11—H11A	109.6	O78—Cl2—O77A	132.9 (5)
C10—C11—H11A	109.6	O89—Cl3—O88	112.8 (4)
N2—C11—H11B	109.6	O89—Cl3—O86	113.2 (4)
C10—C11—H11B	109.6	O88—Cl3—O86	111.0 (3)
H11A—C11—H11B	108.1	O89—Cl3—O87	112.7 (4)
N2—C12—C13	110.4 (3)	O88—Cl3—O87	102.9 (3)
N2—C12—H12A	109.6	O86—Cl3—O87	103.4 (3)
C13—C12—H12A	109.6	O89A—Cl3A—O86A	111.0 (9)
N2—C12—H12B	109.6	O89A—Cl3A—O88A	115.6 (9)
C13—C12—H12B	109.6	O86A—Cl3A—O88A	107.7 (7)
H12A—C12—H12B	108.1	O89A—Cl3A—O87	97.5 (8)
N3—C13—C12	110.2 (3)	O86A—Cl3A—O87	110.8 (6)
N3—C13—H13A	109.6	O88A—Cl3A—O87	114.0 (6)
C12—C13—H13A	109.6	H51W—O51—H51V	116 (10)
N3—C13—H13B	109.6	H52V—O52—H52W	113 (5)
C12—C13—H13B	109.6	H53V—O53—H53W	100 (4)
H13A—C13—H13B	108.1	H54V—O54—H54W	111 (4)
N1—C14—H14A	109.5	H55V—O55—H55W	103 (4)

Hydrogen-bond geometry (Å, °)

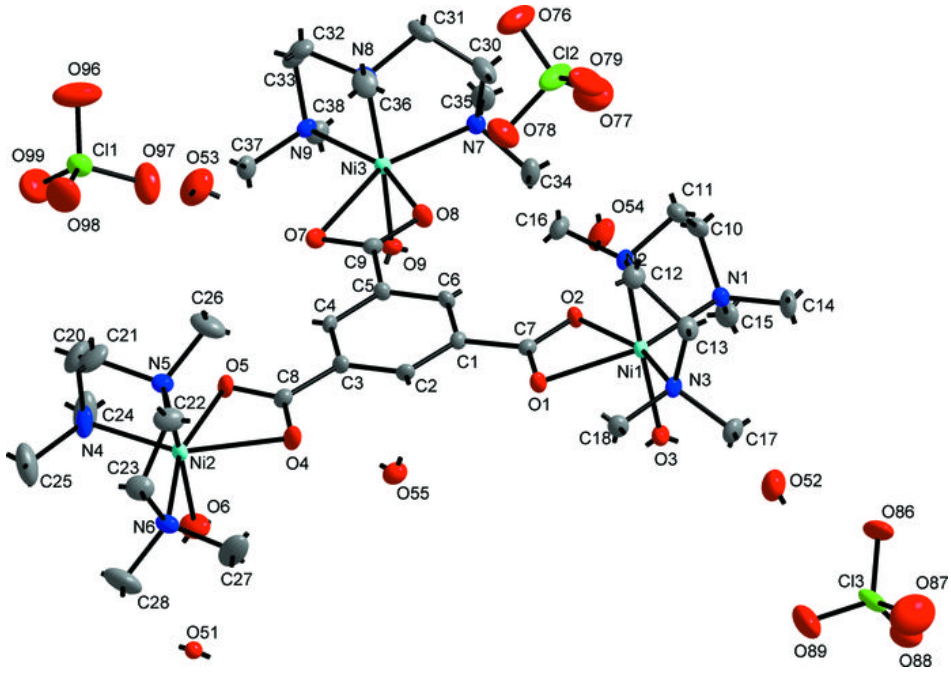
D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3V···O52	0.915 (19)	1.77 (2)	2.677 (4)	173 (4)
O3—H3W···O7^{i}	0.93 (4)	1.83 (2)	2.742 (3)	167 (4)
O6—H6W···O51	0.91 (2)	1.99 (4)	2.607 (9)	124 (4)
O6—H6V···O54^{i}	0.930 (19)	1.83 (2)	2.745 (4)	166 (4)
O9—H9V···O1^{i}	0.908 (19)	1.85 (2)	2.743 (3)	166 (3)
O9—H9W···O55^{i}	0.913 (19)	1.82 (2)	2.727 (3)	171 (4)
O52—H52V···O53^{i}	0.95 (2)	2.13 (5)	2.789 (5)	125 (4)
O53—H53W···O7	0.99 (2)	1.97 (3)	2.877 (4)	151 (5)
O53—H53V···O97	1.00 (2)	1.96 (3)	2.869 (5)	151 (5)
O54—H54V···O2	0.933 (19)	1.98 (2)	2.902 (3)	167 (4)
O55—H55V···O4	0.914 (19)	1.95 (2)	2.843 (3)	166 (4)
O55—H55W···O76^{ii}	0.909 (19)	2.04 (2)	2.934 (4)	168 (4)
C2—H2···O55	0.95	2.69	3.607 (4)	162
C6—H6···O54	0.95	2.67	3.601 (4)	166
C10—H10A···O54	0.99	2.81	3.697 (5)	149

C11—H11B···O79	0.99	2.67	3.477 (11)	139
C11—H11B···O79A	0.99	2.78	3.56 (2)	136
C15—H15A···O54	0.98	2.61	3.392 (5)	137
C15—H15B···O52	0.98	2.66	3.585 (5)	159
C16—H16B···O78	0.98	2.64	3.526 (6)	150
C16—H16B···O79	0.98	2.84	3.396 (9)	117
C17—H17A···O86A	0.98	2.94	3.327 (12)	105
C20—H20B···O98	0.99	2.92	3.310 (6)	105
C21—H21B···O98	0.99	2.77	3.476 (5)	128
C27—H27A···O55	0.98	2.80	3.605 (5)	140
C28—H28A···O51	0.98	2.98	3.941 (10)	169
C30—H30B···O77	0.99	2.76	3.603 (9)	143
C30—H30B···O77A	0.99	2.82	3.513 (15)	128
C30—H30B···O78A	0.99	2.69	3.677 (11)	175
C34—H34B···O54	0.98	2.52	3.293 (5)	136
C36—H36B···O78	0.98	2.66	3.579 (6)	157
C36—H36B···O78A	0.98	2.81	3.783 (11)	171
C37—H37A···O53	0.98	2.85	3.803 (5)	164
C34—H34A···O55^{i}	0.98	2.53	3.487 (5)	165
C37—H37C···O89^{i}	0.98	2.57	3.337 (7)	136
C37—H37C···O89A^{i}	0.98	2.57	3.417 (16)	145
C25—H25B···O88^{iii}	0.98	2.57	3.518 (7)	162
C25—H25B···O88A^{iii}	0.98	2.78	3.704 (12)	157
C24—H24B···O88^{iii}	0.98	2.93	3.797 (7)	148
C24—H24B···O89^{iii}	0.98	2.58	3.439 (9)	146
C15—H15C···O89A^{iv}	0.98	2.93	3.762 (15)	143
C14—H14A···O89A^{iv}	0.98	2.55	3.498 (12)	162
C15—H15C···O89^{iv}	0.98	2.60	3.508 (7)	154
C14—H14A···O89^{iv}	0.98	2.87	3.739 (8)	149
C10—H10B···O87^{iv}	0.99	2.68	3.564 (5)	148
C11—H11A···O97^{ii}	0.99	2.63	3.612 (5)	172
C12—H12A···O78^{ii}	0.99	2.79	3.646 (7)	145
C16—H16A···O78^{ii}	0.98	2.83	3.462 (7)	123
C26—H26C···O79^{ii}	0.98	2.72	3.580 (8)	146
C26—H26C···O79A^{ii}	0.98	2.45	3.276 (14)	142
C14—H14C···O96^{ii}	0.98	2.74	3.423 (5)	127
C14—H14C···O97^{ii}	0.98	2.86	3.754 (5)	153
C18—H18C···O76^{ii}	0.98	2.95	3.809 (5)	147
C13—H13A···O97^{ii}	0.99	2.70	3.562 (5)	146
C17—H17C···O98^{v}	0.98	2.57	3.411 (5)	143
C18—H18A···O98^{v}	0.98	2.88	3.634 (5)	134
C21—H21A···O96^{vi}	0.99	2.87	3.836 (5)	165
C23—H23B···O96^{vi}	0.99	2.84	3.832 (5)	177
C25—H25C···O53^{vi}	0.98	2.61	3.452 (5)	145

C25—H25A···O96^{vi}	0.98	2.44	3.314 (5)	148
C25—H25A···O99^{vi}	0.98	2.94	3.464 (6)	115
C22—H22A···O86A^{vii}	0.99	2.55	3.488 (12)	158
C21—H21B···O86A^{vii}	0.99	2.86	3.395 (13)	115
C26—H26A···O87^{vii}	0.98	2.49	3.405 (6)	154
C22—H22A···O87^{vii}	0.99	2.80	3.512 (5)	130
C28—H28C···O76^{viii}	0.98	2.65	3.614 (5)	169
C35—H35C···O51^{ix}	0.98	2.63	3.390 (9)	134
C30—H30A···O51^{ix}	0.99	2.67	3.565 (9)	151
C33—H33B···O52^{x}	0.99	2.96	3.397 (5)	108
C33—H33B···O86^{x}	0.99	2.37	3.333 (6)	164
C35—H35A···O86^{x}	0.98	2.56	3.510 (6)	163
C35—H35A···O86A^{x}	0.98	2.88	3.756 (12)	149
C33—H33B···O86A^{x}	0.99	2.82	3.689 (11)	147
C33—H33B···O88A^{x}	0.99	2.76	3.715 (12)	162
C33—H33B···O52^{x}	0.99	2.96	3.397 (5)	108
C38—H38B···O86^{x}	0.98	2.81	3.614 (6)	140
C38—H38B···O86A^{x}	0.98	2.57	3.395 (10)	141
C31—H31B···O86^{x}	0.99	2.98	3.960 (7)	170
C31—H31B···O88^{x}	0.99	2.64	3.427 (6)	136
C31—H31B···O88A^{x}	0.99	2.60	3.548 (14)	161
C32—H32A···O99^{xi}	0.99	2.77	3.731 (5)	165
C38—H38C···O96^{xii}	0.98	2.75	3.371 (5)	122

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

Fig. 1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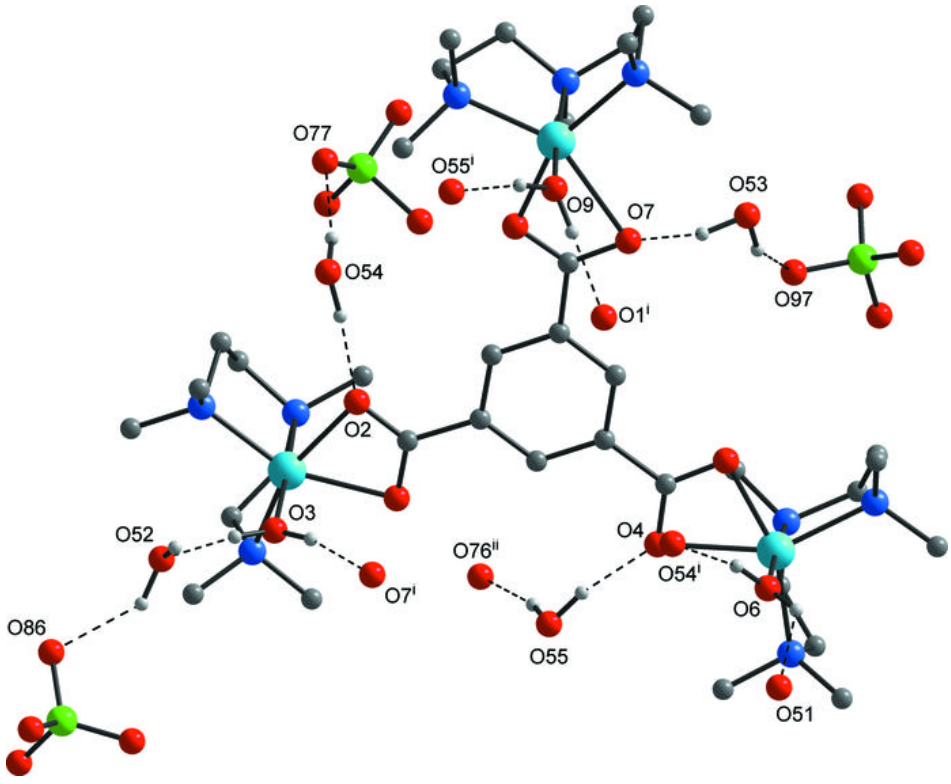
Cl1
O99
O97
O98
O53
C37
N9
C38
C32
C33
N8
C31
C36
C30
O76
Cl2
O79
C35
O77
O78
N7
C34
Ni3
O7
O8
C9
O9
C5
C6
C4
C26
C20
C21
N5
O5
C8
C3
C2
C1
C7
O2
C22
C24
N4
C25
C23
Ni2
O4
O6
N6
C27
C28
O51
O55
C16
O54
C11
C10
N2
C12
N1
C14
C15
C13
Ni1
N3
O1
C18
O3
C17
O52
O86
Cl3
O87
O89
O88

Fig. 2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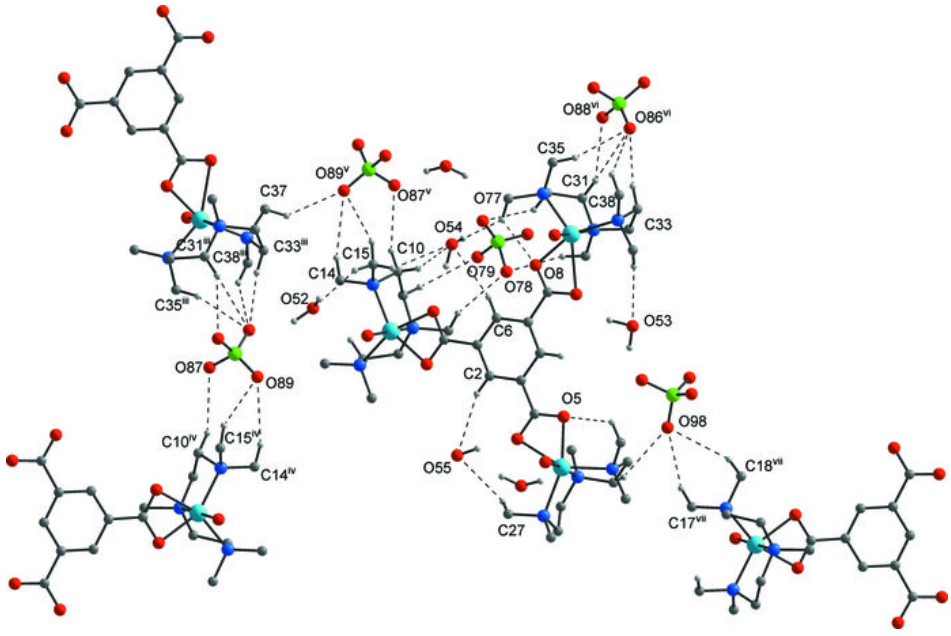
O9
O7
O53
O54
O97
O1^{i}
O2
O3
O52
O7^{i}
O76^{ii}
O4
O54^{i}
O6
O86
O55
O51

Fig. 3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O86^{vi}
C35
C31
C38
C33
O89^{v}
O87^{v}
C37
O77
O54
C15
C10
C14
O79
O8
O78
O52
C6
O53
C31^{iii}
C38^{iii}
C33^{iii}
C35^{iii}
O87
O89
C2
O5
O98
C10^{iv}
C15^{iv}
C14^{iv}
O55
C18^{vii}
C27
C17^{vii}