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{μ-6,6′-Diethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-trinitratosamarium(III)nickel(II)

Rong-Hua Hu,[a] Yan Sui,[a]* Li Chen,[b] Guo-Wei Xie[a] and Yan-Li Ai[a]

[a]JiangXi Province Key Laboratory of Coordination Chemistry, College of Chemistry and Chemical Engineering, JingGangShan University, 343009 Ji'an, JiangXi, People's Republic of China, and [b]College of Education, JingGangShan University, 343009 Ji'an, JiangXi, People's Republic of China
Correspondence e-mail: ysui@163.com

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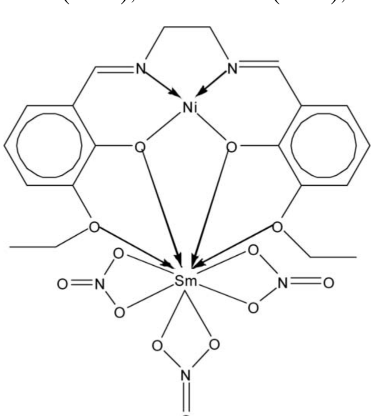
Key indicators: single-crystal X-ray study; *T* = 293 K; mean σ(C–C) = 0.012 Å; *R* factor = 0.045; *wR* factor = 0.084; data-to-parameter ratio = 16.8.

In the title heteronuclear Ni^{II}–Sm^{III} complex (systematic name: {6,6′-diethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato-1$\kappa^4O^1,O^{1\prime},O^6,O^{6\prime}$:2$\kappa^4O^1,N,N'O^{1\prime}$}trinitrato-1$\kappa^6O,O'$-samarium(III)nickel(II)), [NiSm($C_{20}H_{22}N_2O_4$)(NO_3)$_3$], with the hexadentate Schiff base compartmental ligand *N*,*N*′-bis(3-ethoxysalicylidene)ethylenediamine (H_2L) the Ni and Sm atoms are doubly bridged by two phenolate O atoms provided by the Schiff base ligand. The coordination of Ni is square planar with the donor centres of two imine N atoms and two phenolate O atoms. The samarium(III) centre has a decacoordination environment of O atoms, involving the phenolate O atoms, two ethoxy O atoms and two O atoms each from the three nitrates. Some weak C—H···O and O···Ni [3.381 (4) Å] interactions generate a two-dimensional zigzag sheet.

Related literature

For related literature, see: Baggio *et al.* (2000); Caravan *et al.* (1999); Edder *et al.* (2000); Knoer *et al.* (2005); Sui *et al.* (2006).



Experimental

Crystal data

[NiSm($C_{20}H_{22}N_2O_4$)(NO_3)$_3$]
M_r = 749.49
Orthorhombic, $P2_12_12_1$
a = 8.6097 (14) Å
b = 13.750 (2) Å
c = 21.113 (3) Å
V = 2499.4 (7) Å^3
Z = 4
Mo *K*α radiation
μ = 3.16 mm^{-1}
T = 293 (2) K
0.26 × 0.14 × 0.07 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.494, T_{max} = 0.821
18707 measured reflections
6073 independent reflections
2970 reflections with $I > 2\sigma(I)$
R_{int} = 0.089

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.084
S = 1.00
6073 reflections
362 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.58 e Å^{-3}
$\Delta\rho_{min}$ = −1.58 e Å^{-3}
Absolute structure: Flack (1983), 2588 Friedel pairs
Flack parameter: 0.007 (19)

Table 1
Selected bond lengths (Å).

Sm1—O1	2.455 (5)	Sm1—O9	2.507 (6)
Sm1—O2	2.401 (5)	Sm1—O11	2.560 (5)
Sm1—O3	2.621 (5)	Sm1—O12	2.548 (6)
Sm1—O4	2.581 (5)	Ni1—O1	1.861 (5)
Sm1—O5	2.503 (4)	Ni1—O2	1.863 (5)
Sm1—O6	2.573 (5)	Ni1—N1	1.837 (7)
Sm1—O8	2.561 (5)	Ni1—N2	1.840 (6)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C20—H20*A*···O9	0.96	2.49	3.150 (10)	126
C20—H20*C*···O7^{i}	0.96	2.55	3.308 (12)	136
C17—H17*A*···O8^{ii}	0.97	2.59	3.536 (9)	164
C9—H9*A*···O13^{iii}	0.97	2.43	3.283 (11)	147
C7—H7···O13^{iv}	0.93	2.39	3.320 (10)	173

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *APEX2*; software used to prepare material for publication: *APEX2* and *publCIF* (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2499).

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supplementary materials

Acta Cryst. (2007). E**63**, m3191-m3192 [**doi:10.1107/S1600536807061508**]

{μ-6,6'-Diethoxy-2,2'-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}trinitratosamarium(III)nickel(II)

R.-H. Hu, Y. Sui, L. Chen, G.-W. Xie and Y.-L. Ai

Comment

The potential applications of trivalent lanthanide complexes as contrast agent for magnetic resonance imaging and stains for fluorescence imaging have prompted considerable interest in the preparation, magnetic and optical properties of 3 d-4f hetorometallic dinuclear complexes (Baggio *et al.*, 2000; Caravan *et al.*, 1999; Edder *et al.*, 2000; Knoer *et al.*, 2005). As part of our investigations into the structure and applications of 3 d-4f hetorometallic Schiff base complexes (Sui *et al.* 2006), we report here the synthesis and X-ray crystal structure analysis of the title complex, (I), a new Ni^{II}—Sm^{III} complex with salen-type Schiff base *N*,*N*'-bis(3-ethoxysalicylidene) ethylenediamine (H_2L).

Complex (I) crystallizes in the space group $P2_12_12_1$, with nickel and samarium doubly bridged by two phenolate O atoms provided by a salen-type Schiff base ligand. The inner salen-type cavity is occupied by nickel(II), while samarium(III) is present in the open and larger portion of the dinucleating compartmental Schiff base ligand. The dihedral angles between the mean planes of Ni1/O1/O2 and Sm1/O1/O2 is 6.28 (29)° suggesting that the bridging moiety is almost planar, with the deviation of atoms from the least squares Ni1/O1/O2/Sm1 plane being −0.0535 (2)Å for Ni, −0.0354 (3)Å for Sm, 0.0437 (2)Å for O1 and 0.0451 (2)Å for O2.

The samarium(III) center in (I) has a decacoordination environment of O atoms. In addition to the phenolate ligands, two ethoxy O atoms coordinate to this metal center, two O atoms from each of the three nitrates chelate to samarium to complete the decacoordination. The three kinds of Sm—O bond distances are significantly different, the shortest being the Sm—O(phenolate) and longest being the Sm—*O*(ethoxy) separations.

The coordination of nickel(II) is approximately square planar. The donor centers are alternatively above and below the mean N_2O_2 plane with an average deviation from the plane of 0.0798 (2) Å, while Ni1 is 0.0093 (2)Å below this square plane.

Adjacent molecules are held together by weak interactions (O10···Ni1 = 3.381 (4) Å, C7—H7···$O13^{i}$ = 3.320 (10), C9—H9A···$O13^{ii}$ = 3.283 (11), C17—H17A···$O8^{iii}$=3.536 (9) and C20—H20*c*···$O7^{iv}$ = 3.308 (12); symmetry codes: (i) $-x+2, y-1/2, -z+3/2$; (ii) $1+x, y, z$; (iii) $1/2+x, 3/2-y, 2-z$; (iv) $x-1/2, 1/2-y, 2-z$.) these link the molecules into a two-dimensional zigzag sheet (Fig 2).

Experimental

H_2L was prepared by the 2:1 condensation of 3-ethoxysalicylaldehyde and ethylenediamine in methanol. Complex (I) was obtained by the treatment of nickel(II) acetate tetrahydrate (0.217 g, 1 mmol) with H_2L (0.356 g, 1 mmol) in methanol solution (80 ml) under reflux for 3 h and then for another 3 h after the addition of samarium(III) nitrate hexahydrate (0.445 g, 1 mmol). The reaction mixture was cooled and the resulting precipitate was filtered off, washed with diethyl ether and dried *in vacuo*. Single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation at room temperature of

a methanol solution. Analysis calculated for $C_{20}H_{22}N_5NiO_{13}Sm$: C 32.05, H 2.96, N 9.34, Ni 7.83, Sm 20.06%; found: C 32.15, H 2.98, N 9.22, Ni 7.88, Sm 20.22%. IR(KBr, cm^{-1}): 1642(C=N), 1386,1490(nitrate).

Refinement

The H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H distances in the range 0.93 − 0.97 Å, and with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C) for other H atoms.

Figures

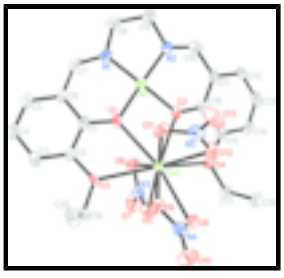

Fig.1. The molecular structure of (I), showing 30% probability displacement ellipsoids. All the H atoms on carbon have been omitted for clarity.

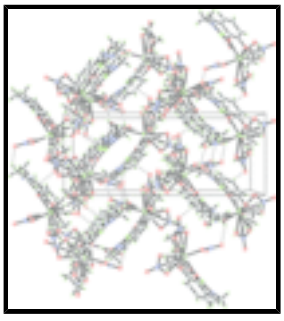

Fig.2. The packing diagram of (I), viewed along the *b* axis; hydrogen bonds are shown as dashed lines.

Table 1. Selected bond lengths(Å).

Table 2 Hydrogen-bond geometry(Å,°).

{6,6'-diethoxy-2,2'-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato-\ 1κ^4*O*^{1},*O*$^{1'}$,*O*^{6},*O*$^{6'}$:2κ^4*O*^{1},*N*,*N*'*O*$^{1'}$}trinitrato-1κ^6*O*,*O*'-\ samarium(III)nickel(II)

Crystal data

$[NiSm(C_{20}H_{22}N_2O_4)(NO_3)_3]$	F_{000} = 1484
M_r = 749.49	D_x = 1.992 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 4344 reflections
a = 8.6097 (14) Å	θ = 1.9–28.2º
b = 13.750 (2) Å	μ = 3.16 mm^{-1}
c = 21.113 (3) Å	*T* = 293 (2) K
V = 2499.4 (7) $Å^3$	Block, red
Z = 4	0.26 × 0.14 × 0.07 mm

Data collection

Bruker APEXII area-detector	6073 independent reflections

diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 0 pixels mm^{-1}

T = 293(2) K

φ and ω scan

Absorption correction: multi-scan (SADABS; Bruker, 2004)

T_{min} = 0.494, T_{max} = 0.821

18707 measured reflections

2970 reflections with $I > 2\sigma(I)$

R_{int} = 0.089

θ_{max} = 28.2º

θ_{min} = 1.9º

$h = -11 \rightarrow 11$

$k = -17 \rightarrow 18$

$l = -28 \rightarrow 27$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.045

$wR(F^2)$ = 0.084

S = 1.00

6073 reflections

362 parameters

1 restraint

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.010P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001

$\Delta\rho_{max}$ = 1.58 e $Å^{-3}$

$\Delta\rho_{min}$ = −1.58 e $Å^{-3}$

Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.00131 (15)

Absolute structure: Flack (1983), 2588 Freidel pairs

Flack parameter: 0.007 (19)

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}
Sm1	0.74822 (5)	0.49875 (3)	0.904649 (15)	0.04039 (13)
Ni1	1.06957 (11)	0.43994 (7)	0.81466 (5)	0.0377 (3)
O2	0.9712 (5)	0.5510 (4)	0.8455 (2)	0.0368 (12)
O1	0.9232 (7)	0.3732 (3)	0.8629 (2)	0.0429 (14)
C16	1.0300 (9)	0.6379 (6)	0.8438 (4)	0.040 (2)

N2	1.1979 (6)	0.5089 (5)	0.7614 (3)	0.0415 (17)
O5	0.7374 (7)	0.5086 (5)	1.0229 (2)	0.0609 (14)
C1	0.9166 (11)	0.2794 (6)	0.8738 (4)	0.039 (2)
O6	0.9601 (6)	0.4659 (4)	0.9879 (3)	0.0578 (18)
O3	0.6921 (6)	0.3156 (4)	0.9304 (2)	0.0418 (15)
N1	1.1761 (8)	0.3307 (5)	0.7897 (3)	0.040 (2)
C11	1.1493 (10)	0.6679 (6)	0.8038 (4)	0.039 (2)
C2	0.7939 (8)	0.2430 (5)	0.9086 (4)	0.039 (2)
C12	1.2037 (9)	0.7641 (6)	0.8051 (4)	0.046 (2)
H12	1.2781	0.7845	0.7761	0.056*
C7	1.1482 (10)	0.2432 (7)	0.8103 (5)	0.050 (3)
H7	1.2178	0.1952	0.7977	0.060*
C19	0.5526 (9)	0.2857 (6)	0.9639 (4)	0.047 (2)
H19A	0.5074	0.3418	0.9848	0.057*
H19B	0.5803	0.2387	0.9962	0.057*
C6	1.0246 (9)	0.2123 (5)	0.8494 (4)	0.0364 (19)
O4	0.8532 (6)	0.6719 (4)	0.9247 (2)	0.0404 (14)
C15	0.9653 (10)	0.7080 (6)	0.8862 (4)	0.037 (2)
C4	0.8980 (11)	0.0817 (6)	0.9028 (4)	0.054 (2)
H4	0.8940	0.0164	0.9142	0.064*
N3	0.8787 (9)	0.4933 (6)	1.0341 (3)	0.0523 (17)
C10	1.2268 (11)	0.5986 (6)	0.7611 (4)	0.048 (2)
H10	1.3003	0.6221	0.7327	0.058*
O7	0.9325 (7)	0.4984 (5)	1.0867 (3)	0.0793 (17)
C20	0.4353 (10)	0.2419 (6)	0.9207 (4)	0.064 (3)
H20A	0.4092	0.2877	0.8881	0.096*
H20B	0.3437	0.2256	0.9444	0.096*
H20C	0.4774	0.1841	0.9018	0.096*
C5	1.0135 (10)	0.1148 (6)	0.8660 (4)	0.048 (2)
H5	1.0880	0.0713	0.8512	0.058*
C13	1.1476 (11)	0.8275 (6)	0.8488 (4)	0.058 (2)
H13	1.1907	0.8893	0.8519	0.070*
O11	0.6799 (7)	0.4591 (5)	0.7895 (3)	0.0548 (19)
O9	0.4640 (7)	0.4655 (4)	0.8904 (3)	0.0610 (18)
N5	0.6398 (9)	0.5460 (7)	0.7739 (4)	0.054 (2)
O12	0.6472 (7)	0.6093 (4)	0.8170 (3)	0.0569 (16)
O8	0.5117 (7)	0.5881 (5)	0.9497 (3)	0.0611 (19)
N4	0.4132 (9)	0.5368 (6)	0.9234 (4)	0.053 (2)
O10	0.2729 (8)	0.5498 (6)	0.9315 (3)	0.083 (2)
C17	0.7801 (9)	0.7381 (5)	0.9708 (3)	0.043 (2)
H17A	0.8603	0.7760	0.9916	0.051*
H17B	0.7275	0.6999	1.0029	0.051*
C3	0.7828 (10)	0.1467 (6)	0.9240 (4)	0.045 (2)
H3	0.7001	0.1243	0.9483	0.054*
C8	1.3125 (10)	0.3502 (7)	0.7503 (4)	0.055 (3)
H8A	1.3308	0.2971	0.7211	0.066*
H8B	1.4043	0.3589	0.7764	0.066*
C9	1.2742 (10)	0.4432 (6)	0.7147 (3)	0.050 (2)
H9A	1.3680	0.4728	0.6982	0.060*

H9B	1.2047	0.4296	0.6797	0.060*
C14	1.0292 (9)	0.8018 (6)	0.8880 (4)	0.047 (2)
H14	0.9899	0.8472	0.9164	0.057*
O13	0.5995 (7)	0.5672 (5)	0.7215 (3)	0.083 (2)
C18	0.6670 (10)	0.8050 (6)	0.9409 (4)	0.058 (3)
H18A	0.7186	0.8434	0.9093	0.087*
H18B	0.6235	0.8470	0.9726	0.087*
H18C	0.5854	0.7679	0.9214	0.087*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Sm1	0.0467 (2)	0.03015 (18)	0.04428 (19)	0.0004 (3)	0.0029 (3)	−0.0022 (2)
Ni1	0.0425 (6)	0.0292 (5)	0.0414 (5)	−0.0002 (5)	0.0048 (5)	−0.0048 (5)
O2	0.043 (3)	0.023 (3)	0.045 (3)	−0.003 (3)	0.005 (3)	0.002 (3)
O1	0.062 (4)	0.021 (3)	0.046 (3)	−0.004 (3)	0.007 (3)	0.002 (2)
C16	0.049 (5)	0.041 (5)	0.030 (4)	−0.006 (4)	−0.002 (4)	−0.008 (4)
N2	0.050 (4)	0.028 (4)	0.047 (4)	−0.008 (4)	0.005 (3)	−0.004 (4)
O5	0.075 (4)	0.071 (4)	0.037 (3)	0.007 (6)	0.011 (3)	0.009 (3)
C1	0.040 (5)	0.025 (5)	0.051 (5)	−0.004 (4)	−0.007 (5)	−0.007 (4)
O6	0.044 (3)	0.075 (5)	0.054 (4)	0.008 (3)	0.002 (3)	0.005 (3)
O3	0.051 (4)	0.027 (3)	0.048 (3)	−0.009 (3)	0.015 (3)	0.008 (2)
N1	0.044 (5)	0.034 (4)	0.043 (5)	0.003 (4)	0.005 (4)	−0.011 (4)
C11	0.048 (6)	0.026 (5)	0.044 (6)	0.000 (4)	0.000 (5)	0.000 (4)
C2	0.047 (6)	0.024 (4)	0.044 (5)	0.000 (4)	−0.007 (4)	0.000 (4)
C12	0.048 (6)	0.036 (5)	0.055 (6)	−0.011 (4)	0.013 (4)	0.000 (4)
C7	0.044 (6)	0.042 (6)	0.064 (6)	0.018 (5)	0.002 (5)	−0.024 (5)
C19	0.044 (5)	0.047 (5)	0.052 (6)	−0.006 (5)	0.004 (5)	0.005 (4)
C6	0.039 (5)	0.025 (4)	0.045 (5)	0.007 (4)	−0.007 (5)	−0.003 (4)
O4	0.054 (4)	0.026 (3)	0.041 (3)	−0.001 (3)	0.008 (3)	−0.010 (2)
C15	0.046 (6)	0.028 (5)	0.039 (5)	0.002 (4)	−0.006 (4)	−0.004 (4)
C4	0.077 (7)	0.027 (5)	0.057 (5)	0.006 (5)	−0.004 (5)	0.005 (4)
N3	0.065 (5)	0.043 (4)	0.049 (5)	−0.005 (5)	0.001 (4)	0.011 (5)
C10	0.042 (6)	0.064 (6)	0.038 (5)	−0.012 (5)	0.000 (5)	0.001 (4)
O7	0.120 (5)	0.069 (4)	0.049 (4)	0.011 (6)	−0.026 (4)	0.002 (5)
C20	0.057 (6)	0.057 (6)	0.078 (7)	−0.018 (6)	−0.014 (6)	0.019 (5)
C5	0.055 (6)	0.034 (5)	0.055 (6)	0.006 (4)	−0.001 (5)	−0.002 (4)
C13	0.074 (6)	0.029 (5)	0.072 (6)	−0.024 (5)	0.003 (6)	0.002 (5)
O11	0.066 (4)	0.058 (4)	0.040 (4)	0.013 (4)	0.000 (3)	−0.021 (3)
O9	0.068 (4)	0.037 (4)	0.078 (5)	0.003 (3)	0.013 (4)	−0.012 (3)
N5	0.043 (5)	0.065 (6)	0.053 (6)	−0.007 (5)	−0.004 (4)	−0.004 (5)
O12	0.070 (4)	0.041 (4)	0.060 (4)	−0.008 (3)	−0.014 (4)	0.001 (3)
O8	0.056 (4)	0.058 (5)	0.069 (4)	0.017 (3)	−0.004 (3)	−0.025 (3)
N4	0.039 (5)	0.060 (6)	0.059 (5)	−0.006 (4)	−0.001 (4)	0.015 (4)
O10	0.052 (4)	0.099 (5)	0.098 (5)	0.009 (5)	0.003 (4)	−0.013 (4)
C17	0.059 (7)	0.032 (4)	0.038 (5)	0.006 (5)	0.006 (4)	−0.011 (3)
C3	0.050 (7)	0.033 (5)	0.051 (5)	−0.012 (5)	−0.009 (4)	0.003 (3)
C8	0.045 (6)	0.061 (7)	0.060 (6)	0.005 (5)	0.010 (5)	−0.014 (5)

C9	0.061 (6)	0.044 (5)	0.045 (4)	0.001 (5)	−0.001 (5)	−0.009 (4)
C14	0.043 (6)	0.034 (5)	0.065 (7)	0.001 (4)	−0.006 (5)	−0.016 (4)
O13	0.097 (5)	0.106 (5)	0.045 (4)	−0.039 (5)	−0.024 (4)	0.023 (4)
C18	0.062 (6)	0.037 (5)	0.076 (7)	0.008 (5)	0.003 (5)	0.003 (5)

Geometric parameters (Å, °)

Sm1—O1	2.455 (5)	C19—H19A	0.9700
Sm1—O2	2.401 (5)	C19—H19B	0.9700
Sm1—O3	2.621 (5)	C6—C5	1.390 (10)
Sm1—O4	2.581 (5)	O4—C15	1.356 (9)
Sm1—O5	2.503 (4)	O4—C17	1.473 (8)
Sm1—O6	2.573 (5)	C15—C14	1.404 (11)
Sm1—O8	2.561 (5)	C4—C5	1.341 (10)
Sm1—O9	2.507 (6)	C4—C3	1.408 (10)
Sm1—O11	2.560 (5)	C4—H4	0.9300
Sm1—O12	2.548 (6)	N3—O7	1.205 (7)
Ni1—O1	1.861 (5)	C10—H10	0.9300
Ni1—O2	1.863 (5)	C20—H20A	0.9600
Ni1—N1	1.837 (7)	C20—H20B	0.9600
Ni1—N2	1.840 (6)	C20—H20C	0.9600
O2—C16	1.298 (9)	C5—H5	0.9300
O1—C1	1.312 (8)	C13—C14	1.359 (11)
C16—C11	1.391 (10)	C13—H13	0.9300
C16—C15	1.429 (10)	O11—N5	1.287 (9)
N2—C10	1.257 (9)	O9—N4	1.279 (9)
N2—C9	1.490 (9)	N5—O13	1.195 (9)
O5—N3	1.257 (8)	N5—O12	1.261 (9)
C1—C2	1.381 (10)	O8—N4	1.235 (8)
C1—C6	1.407 (11)	N4—O10	1.233 (9)
O6—N3	1.259 (7)	C17—C18	1.481 (10)
O3—C2	1.405 (8)	C17—H17A	0.9700
O3—C19	1.453 (9)	C17—H17B	0.9700
N1—C7	1.303 (10)	C3—H3	0.9300
N1—C8	1.463 (10)	C8—C9	1.519 (11)
C11—C12	1.404 (10)	C8—H8A	0.9700
C11—C10	1.472 (11)	C8—H8B	0.9700
C2—C3	1.368 (10)	C9—H9A	0.9700
C12—C13	1.357 (11)	C9—H9B	0.9700
C12—H12	0.9300	C14—H14	0.9300
C7—C6	1.412 (11)	C18—H18A	0.9600
C7—H7	0.9300	C18—H18B	0.9600
C19—C20	1.488 (11)	C18—H18C	0.9600
O2—Sm1—O1	62.13 (17)	C13—C12—H12	120.0
O2—Sm1—O5	122.20 (18)	C11—C12—H12	120.0
O1—Sm1—O5	114.80 (19)	N1—C7—C6	127.7 (8)
O2—Sm1—O9	140.61 (17)	N1—C7—H7	116.1
O1—Sm1—O9	115.33 (19)	C6—C7—H7	116.1
O5—Sm1—O9	95.3 (2)	O3—C19—C20	112.2 (7)

O2—Sm1—O12	73.54 (18)	O3—C19—H19A	109.2
O1—Sm1—O12	111.58 (17)	C20—C19—H19A	109.2
O5—Sm1—O12	132.8 (2)	O3—C19—H19B	109.2
O9—Sm1—O12	71.85 (18)	C20—C19—H19B	109.2
O2—Sm1—O11	75.72 (17)	H19A—C19—H19B	107.9
O1—Sm1—O11	69.51 (19)	C5—C6—C1	119.7 (8)
O5—Sm1—O11	161.9 (2)	C5—C6—C7	119.3 (7)
O9—Sm1—O11	67.87 (19)	C1—C6—C7	121.0 (7)
O12—Sm1—O11	50.1 (2)	C15—O4—C17	118.3 (6)
O2—Sm1—O8	133.3 (2)	C15—O4—Sm1	119.2 (4)
O1—Sm1—O8	163.45 (19)	C17—O4—Sm1	121.9 (4)
O5—Sm1—O8	64.8 (2)	O4—C15—C14	126.9 (8)
O9—Sm1—O8	49.90 (19)	O4—C15—C16	114.0 (7)
O12—Sm1—O8	73.3 (2)	C14—C15—C16	118.9 (8)
O11—Sm1—O8	105.78 (19)	C5—C4—C3	119.4 (7)
O2—Sm1—O6	80.85 (17)	C5—C4—H4	120.3
O1—Sm1—O6	71.76 (18)	C3—C4—H4	120.3
O5—Sm1—O6	49.78 (18)	O7—N3—O5	122.4 (7)
O9—Sm1—O6	137.87 (18)	O7—N3—O6	121.2 (8)
O12—Sm1—O6	147.46 (18)	O5—N3—O6	116.3 (7)
O11—Sm1—O6	140.73 (19)	N2—C10—C11	122.9 (8)
O8—Sm1—O6	113.23 (19)	N2—C10—H10	118.6
O2—Sm1—O4	61.93 (16)	C11—C10—H10	118.6
O1—Sm1—O4	119.50 (17)	C19—C20—H20A	109.5
O5—Sm1—O4	78.42 (18)	C19—C20—H20B	109.5
O9—Sm1—O4	121.96 (18)	H20A—C20—H20B	109.5
O12—Sm1—O4	71.87 (17)	C19—C20—H20C	109.5
O11—Sm1—O4	115.66 (19)	H20A—C20—H20C	109.5
O8—Sm1—O4	77.00 (19)	H20B—C20—H20C	109.5
O6—Sm1—O4	78.55 (17)	C4—C5—C6	121.6 (8)
O2—Sm1—O3	122.88 (16)	C4—C5—H5	119.2
O1—Sm1—O3	60.80 (16)	C6—C5—H5	119.2
O5—Sm1—O3	80.69 (18)	C12—C13—C14	120.9 (8)
O9—Sm1—O3	70.72 (17)	C12—C13—H13	119.5
O12—Sm1—O3	131.34 (17)	C14—C13—H13	119.5
O11—Sm1—O3	87.11 (19)	N5—O11—Sm1	96.2 (5)
O8—Sm1—O3	103.73 (19)	N4—O9—Sm1	97.4 (5)
O6—Sm1—O3	79.66 (17)	O13—N5—O12	120.9 (10)
O4—Sm1—O3	156.43 (16)	O13—N5—O11	122.8 (9)
N1—Ni1—N2	86.9 (3)	O12—N5—O11	116.2 (8)
N1—Ni1—O1	95.3 (3)	N5—O12—Sm1	97.5 (5)
N2—Ni1—O1	174.1 (3)	N4—O8—Sm1	96.0 (5)
N1—Ni1—O2	175.6 (3)	O10—N4—O8	121.8 (9)
N2—Ni1—O2	93.7 (3)	O10—N4—O9	121.5 (9)
O1—Ni1—O2	84.6 (2)	O8—N4—O9	116.6 (8)
C16—O2—Ni1	124.6 (5)	O4—C17—C18	112.5 (6)
C16—O2—Sm1	127.0 (5)	O4—C17—H17A	109.1
Ni1—O2—Sm1	107.4 (2)	C18—C17—H17A	109.1
C1—O1—Ni1	127.6 (6)	O4—C17—H17B	109.1

C1—O1—Sm1	127.0 (5)	C18—C17—H17B	109.1
Ni1—O1—Sm1	105.4 (2)	H17A—C17—H17B	107.8
O2—C16—C11	125.3 (7)	C2—C3—C4	119.3 (7)
O2—C16—C15	116.8 (7)	C2—C3—H3	120.3
C11—C16—C15	117.9 (7)	C4—C3—H3	120.3
C10—N2—C9	120.3 (7)	N1—C8—C9	105.1 (7)
C10—N2—Ni1	128.8 (6)	N1—C8—H8A	110.7
C9—N2—Ni1	110.8 (5)	C9—C8—H8A	110.7
N3—O5—Sm1	98.2 (4)	N1—C8—H8B	110.7
O1—C1—C2	118.9 (8)	C9—C8—H8B	110.7
O1—C1—C6	123.5 (8)	H8A—C8—H8B	108.8
C2—C1—C6	117.6 (7)	N2—C9—C8	106.2 (6)
N3—O6—Sm1	94.7 (4)	N2—C9—H9A	110.5
C2—O3—C19	118.3 (6)	C8—C9—H9A	110.5
C2—O3—Sm1	119.9 (4)	N2—C9—H9B	110.5
C19—O3—Sm1	121.7 (4)	C8—C9—H9B	110.5
C7—N1—C8	120.5 (7)	H9A—C9—H9B	108.7
C7—N1—Ni1	124.6 (6)	C13—C14—C15	121.1 (8)
C8—N1—Ni1	114.5 (6)	C13—C14—H14	119.4
C16—C11—C12	120.9 (8)	C15—C14—H14	119.4
C16—C11—C10	120.9 (7)	C17—C18—H18A	109.5
C12—C11—C10	118.1 (8)	C17—C18—H18B	109.5
C3—C2—C1	122.1 (7)	H18A—C18—H18B	109.5
C3—C2—O3	124.5 (7)	C17—C18—H18C	109.5
C1—C2—O3	113.2 (6)	H18A—C18—H18C	109.5
C13—C12—C11	120.0 (8)	H18B—C18—H18C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C20—H20A···O9	0.96	2.49	3.150 (10)	126
C20—H20C···O7^{i}	0.96	2.55	3.308 (12)	136
C17—H17A···O8^{ii}	0.97	2.59	3.536 (9)	164
C9—H9A···O13^{iii}	0.97	2.43	3.283 (11)	147
C7—H7···O13^{iv}	0.93	2.39	3.320 (10)	173

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

Fig. 1

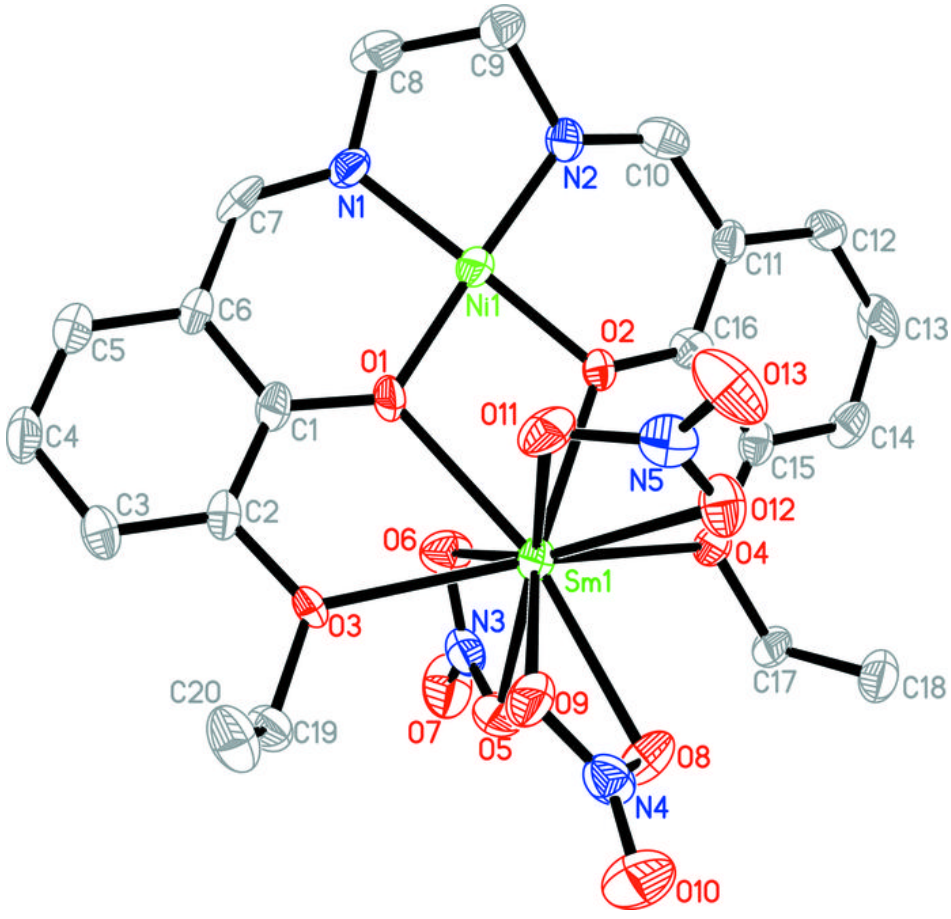
C8
C9
N2
C10
N1
C7
C12
C11
Ni1
C6
C13
C5
O2
C16
O1
O13
O11
C4
C1
C14
N5
C15
C3
C2
O12
O6
O4
Sm1
O3
N3
C17
C18
C20
O9
C19
O7
O5
O8
N4
O10

Fig. 2