Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

{6,6′-Diethoxy-2,2′-[ethane-1,2-diyl-bis(nitrilomethylidyne)]diphenolato}-trinitratoyttrium(III)nickel(II)

Yan Sui,[a]* Qiang Huang,[b] Rong-Hua Hu[a] and Guo-Wei Xie[c]

[a]College of Chemistry and Chemical Engineering, JingGangShan University, 343009 Ji'an, JiangXi, People's Republic of China, [b]College of Mechanical Engineering, Jiujiang University, 332005 JiuJiang, JiangXi, People's Republic of China, and [c]College of Education, JingGangShan University, 343009 Ji'an, JiangXi, People's Republic of China
Correspondence e-mail: ysui@163.com

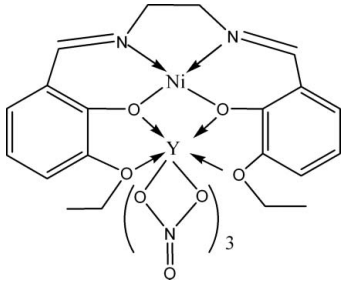
Received 22 June 2007; accepted 25 June 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.028; wR factor = 0.061; data-to-parameter ratio = 16.5.

The title heteronuclear Ni^{II}–Y^{III} complex (systematic name: {6,6′-diethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]-diphenolato-1$\kappa^4O^1,O^{1'},O^6,O^{6'}$:2$\kappa^4O^1,N,N',O^{1'}$}trinitrato-1$\kappa^6O,O'$-yttrium(III)nickel(II)), $[NiY(C_{20}H_{22}N_2O_4)(NO_3)_3]$, with the hexadentate Schiff base compartmental ligand N,N'-bis(3-ethoxysalicylidene)ethylenediamine (H_2L), has been synthesized and structurally characterized. The Ni and Y atoms are doubly bridged by two phenolate O atoms provided by the Schiff base ligand. The coordination of the Ni atom is square planar, formed by two imine N atoms and two phenolate O atoms. The Y^{III} atom has a decacoordination environment of O atoms, formed by the phenolate O atoms, two ethoxy O atoms and two O atoms each from three nitrate ligands. No classical intermolecular hydrogen bonds are found. Some weak C—H···O and O···Ni interactions generate a two-dimensional zigzag sheet.

Related literature

For related literature, see: Baggio *et al.* (2000); Brewer *et al.* (2001); Caravan *et al.* (1999); Edder *et al.* (2000); Mohanta *et al.* (2002); Wong *et al.* (2002).



Experimental

Crystal data

$[NiY(C_{20}H_{22}N_2O_4)(NO_3)_3]$
M_r = 688.05
Orthorhombic, $P2_12_12_1$
a = 8.5837 (10) Å
b = 13.7547 (16) Å
c = 21.153 (2) Å
V = 2497.5 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.15 mm^{-1}
T = 293 (2) K
0.32 × 0.28 × 0.19 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.380, T_{max} = 0.559
18612 measured reflections
5977 independent reflections
4933 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.061
S = 0.98
5977 reflections
363 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.56 e Å^{-3}
Absolute structure: Flack (1983), 2490Friedel pairs
Flack parameter: −0.011 (5)

Table 1
Selected geometric parameters (Å, °).

Y1—O1	2.3073 (17)	Y1—O10	2.454 (2)
Y1—O2	2.3602 (18)	Y1—O12	2.6424 (17)
Y1—O3	2.472 (2)	Y1—O13	2.5708 (17)
Y1—O5	2.451 (2)	Ni1—O1	1.8938 (17)
Y1—O6	2.409 (2)	Ni1—O2	1.8927 (18)
Y1—O7	2.560 (2)	Ni1—N1	1.907 (2)
Y1—O9	2.399 (2)	Ni1—N2	1.908 (2)
O1—Y1—O2	65.69 (6)	O6—Y1—O5	73.16 (7)
O1—Y1—O3	75.50 (7)	O6—Y1—O7	50.55 (7)
O1—Y1—O5	74.16 (7)	O6—Y1—O10	136.94 (8)
O1—Y1—O6	142.25 (7)	O6—Y1—O12	68.19 (6)
O1—Y1—O7	130.85 (8)	O6—Y1—O13	121.83 (7)
O1—Y1—O9	124.85 (7)	O7—Y1—O12	102.84 (7)
O1—Y1—O10	80.25 (7)	O7—Y1—O13	74.42 (7)
O1—Y1—O12	126.25 (6)	O9—Y1—O3	159.49 (8)
O1—Y1—O13	62.86 (6)	O9—Y1—O5	130.82 (8)
O2—Y1—O3	69.65 (7)	O9—Y1—O6	91.16 (8)
O2—Y1—O5	115.01 (6)	O9—Y1—O7	64.52 (8)
O2—Y1—O6	113.00 (7)	O9—Y1—O10	52.16 (7)
O2—Y1—O7	162.14 (7)	O9—Y1—O12	76.33 (7)
O2—Y1—O9	114.04 (7)	O9—Y1—O13	79.21 (7)
O2—Y1—O10	72.17 (7)	O10—Y1—O3	140.57 (8)
O2—Y1—O12	60.71 (6)	O10—Y1—O7	113.97 (7)
O2—Y1—O13	123.36 (6)	O10—Y1—O12	80.31 (7)
O3—Y1—O7	105.42 (8)	O10—Y1—O13	77.38 (7)
O3—Y1—O12	89.47 (7)	O13—Y1—O12	153.82 (7)
O3—Y1—O13	116.54 (7)	O1—Ni1—N1	94.30 (8)
O5—Y1—O3	51.71 (7)	O1—Ni1—N2	177.80 (9)
O5—Y1—O7	69.98 (8)	O2—Ni1—O1	83.93 (7)
O5—Y1—O10	146.17 (7)	O2—Ni1—N1	172.48 (10)
O5—Y1—O12	132.95 (6)	O2—Ni1—N2	95.83 (9)
O5—Y1—O13	71.44 (6)	N1—Ni1—N2	86.22 (10)
O6—Y1—O3	69.61 (8)		

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *APEX2*; program(s) used to refine structure: *APEX2*; molecular graphics: *APEX2*; software used to prepare material for publication: *APEX2*.

The authors gratefully acknowledge financial support from the Department of Education, JiangXi Province (grant Nos. 2007-317 and 05YB195).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2246).

References

Baggio, R., Garland, M. T., Moreno, Y., Pena, O., Perec, M. & Spodine, E. (2000). *J. Chem. Soc. Dalton Trans.* pp. 2061–2066.

Brewer, C., Brewer, G., Scheidt, W. R., Shang, M. & Carpenter, E. E. (2001). *Inorg. Chim. Acta*, **313**, 65–70.

Bruker (2004). *APEX2*. Version 1.22. Bruker AXS Inc., Madison, Wisconsin, USA.

Caravan, P., Ellison, J. J., McMurry, T. J. & Lauffer, R. B. (1999). *Chem. Rev.* **99**, 2293–2352.

Edder, C., Piguet, C., Bernardinelli, G., Mareda, J., Bochet, C. G., Bunzli, J.-C.G. & Hopfgartner, G. (2000). *Inorg. Chem.* **39**, 5059–5073.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Mohanta, S., Lin, H.-H., Lee, C.-J. & Wei, H.-H. (2002). *Inorg. Chem. Commun.* **5**, 585–588.

Wong, W.-K., Liang, H., Wong, W.-Y., Cai, Z., Li, K.-F. & Cheah, K. W. (2002). *New J. Chem.* **26**, 275–578.

supplementary materials

Acta Cryst. (2007). E**63**, m2015-m2016 [**doi:10.1107/S1600536807030875**]

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

Y. Sui, Q. Huang, R.-H. Hu and G.-W. Xie

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 heterometallic dinuclear complexes (Baggio *et al.*, 2000; Caravan *et al.*, 1999; Edder *et al.*, 2000). Recently, some 3 d-4f heterometallic Schiff base complexes have been synthesized, such as Cu^{II}—Gd^{III}, Ni^{II}—Gd^{III} and Zn^{II}—Ho^{III} heterodinuclear complexes (Brewer *et al.*, 2001; Mohanta *et al.*, 2002; Wong *et al.*, 2002), which exhibits novel magnetic and luminescent properties, As part of our investigations into the structure and applications of 3 d-4f hetorometallic Schiff base complexes, we report here the synthesis and X-ray crystal structure analysis of the title complex, (I), a new Ni^{II}—Y^{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 yttrium 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 yttrium(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 Y1/O1/O2 is 4.1 (2)° suggesting that the bridging moiety is almost planar; the deviation of atoms from the least squares Ni1/O1/O2/Y1 plane being −0.0340 (3)Å for Ni, −0.0244 (2)Å for Y, 0.096 (3)Å for O1 and 0.0287 (3)Å for O2.

The yttrium(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 each from the three nitrates chelate to yttrium to complete the decacoordination. The three kinds of Y—O bond distances are significantly different, the shortest being the Y—O(phenolate) and longest being the *Y*—*O*(ethoxy) separations.

The coordination of nickel(II) is square planar. The donor centers are alternatively above and below the mean N_2O_2 plane with an average deviation from the plane of 0.0782 (2) Å, while Ni1 is 0.0423 (3)Å above this square plane.

Adjacent molecules are held together by weak interactions (O8···Ni1 = 3.146 (4) Å, C9—H9B···$O4^{i}$ = 3.278 (4) and C7—H7···$O4^{ii}$ = 3.293 (4); symmetry codes:(i)*x* − 1, *y*, *z*; (ii)-*x*, *y* − 1/2, 3/2 − *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 (50 ml) under reflux for 3 h and then for another 3 h after the addition of yttrium(III) nitrate hexahydrate (0.383 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 $C_{20}H_{22}N_5NiO_{13}Y$: C 31.94, H 3.22, N 10.18, Ni 8.53, Y 12.92%; found: C 31.92, H 3.15, N 10.20, Ni 8.50, Y 12.90%. IR (KBr, cm^{-1}): 1640 (C=N), 1384, 1491 (nitrate).

Refinement

The H atoms were positioned geometrically and treated as riding on their parent atoms, with C—H distances of 0.97 (methylene) and 0.96 Å (methyl), and with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl H atoms and $1.2U_{eq}(C)$ for other H atoms.

Figures

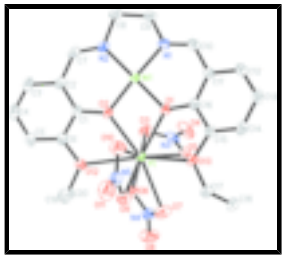


Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids. All the H atoms on carbon have been omitted for clarity.

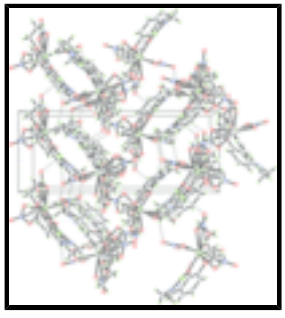


Fig. 2. The packing diagram of (I), viewed along the *b* axis; hydrogen bonds are shown as dashed lines.

Table 1. Selected geometric parameters (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*'- yttrium(III)nickel(II)

Crystal data

$[NiY(C_{20}H_{22}N_2O_4)(NO_3)_3]$	$F_{000} = 1392$
$M_r = 688.05$	$D_x = 1.830$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 18612 reflections
$a = 8.5837$ (10) Å	θ = 1.8–28.2°
$b = 13.7547$ (16) Å	μ = 3.15 mm^{-1}
$c = 21.153$ (2) Å	$T = 293$ (2) K
$V = 2497.5$ (5) Å^3	Block, red
$Z = 4$	0.32 × 0.28 × 0.19 mm

Data collection

Bruker APEXII area-detector diffractometer	5977 independent reflections
Radiation source: fine-focus sealed tube	4933 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.028$
Detector resolution: 0 pixels mm^{-1}	$\theta_{max} = 28.2^o$
$T = 293(2)$ K	$\theta_{min} = 1.8^o$
φ and ω scans	$h = -10 \rightarrow 11$
Absorption correction: multi-scan (SADABS; Bruker, 2004)	$k = -18 \rightarrow 18$
$T_{min} = 0.380$, $T_{max} = 0.559$	$l = -28 \rightarrow 27$
18612 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.028$	$w = 1/[\sigma^2(F_o^2) + (0.0001P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.061$	$(\Delta/\sigma)_{max} = 0.003$
$S = 0.98$	$\Delta\rho_{max} = 0.37$ e Å^{-3}
5977 reflections	$\Delta\rho_{min} = -0.56$ e Å^{-3}
363 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), with how many Friedel pairs?
Secondary atom site location: difference Fourier map	Flack parameter: −0.011 (5)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Y1	0.25957 (3)	1.001598 (17)	0.899553 (11)	0.03377 (7)
Ni1	−0.06212 (4)	0.94357 (2)	0.816733 (16)	0.03396 (8)
O1	0.0443 (2)	1.05622 (12)	0.84475 (8)	0.0363 (4)

C10	−0.2219 (4)	1.1056 (2)	0.76256 (12)	0.0421 (7)
H10	−0.2938	1.1313	0.7342	0.051*
C16	−0.0176 (3)	1.14513 (16)	0.84431 (12)	0.0326 (6)
O13	0.1656 (2)	1.17532 (12)	0.92318 (8)	0.0390 (4)
C15	0.0455 (3)	1.21262 (17)	0.88636 (12)	0.0342 (6)
C2	−0.0145 (3)	0.71544 (18)	0.84982 (13)	0.0381 (6)
C11	−0.1426 (3)	1.17201 (19)	0.80476 (13)	0.0373 (6)
C7	−0.1416 (3)	0.74305 (19)	0.80946 (14)	0.0435 (7)
H7	−0.2064	0.6936	0.7951	0.052*
C1	0.0975 (3)	0.78135 (17)	0.87331 (12)	0.0349 (6)
C6	0.2204 (3)	0.74670 (17)	0.90983 (12)	0.0362 (6)
C17	0.2364 (4)	1.23942 (19)	0.97003 (12)	0.0447 (7)
H17A	0.2896	1.2004	1.0015	0.054*
H17B	0.1551	1.2759	0.9913	0.054*
C4	0.1187 (4)	0.58477 (19)	0.90364 (15)	0.0527 (8)
H4	0.1246	0.5194	0.9146	0.063*
C3	−0.0007 (4)	0.61647 (19)	0.86702 (14)	0.0487 (8)
H3	−0.0749	0.5722	0.8529	0.058*
C18	0.3505 (4)	1.3092 (2)	0.94085 (16)	0.0582 (9)
H18A	0.2967	1.3515	0.9121	0.087*
H18B	0.4290	1.2735	0.9184	0.087*
H18C	0.3987	1.3472	0.9735	0.087*
C13	−0.1360 (4)	1.3332 (2)	0.85042 (15)	0.0509 (8)
H13	−0.1756	1.3960	0.8526	0.061*
C12	−0.1973 (4)	1.2684 (2)	0.80845 (15)	0.0484 (8)
H12	−0.2771	1.2883	0.7816	0.058*
C14	−0.0143 (4)	1.30548 (18)	0.89000 (14)	0.0442 (7)
H14	0.0267	1.3496	0.9189	0.053*
O9	0.2877 (3)	1.00638 (16)	1.01235 (9)	0.0548 (5)
O5	0.3602 (2)	1.11027 (13)	0.81731 (10)	0.0498 (5)
O2	0.0936 (2)	0.87691 (12)	0.86291 (9)	0.0411 (5)
N1	−0.1987 (3)	1.01332 (16)	0.76193 (10)	0.0402 (5)
O10	0.0577 (3)	0.97519 (16)	0.97990 (10)	0.0596 (6)
O12	0.3210 (2)	0.81926 (12)	0.92998 (9)	0.0408 (5)
N5	0.1451 (3)	0.99281 (18)	1.02646 (12)	0.0489 (6)
O3	0.3174 (3)	0.96332 (15)	0.78781 (10)	0.0537 (6)
N3	0.3590 (3)	1.0473 (2)	0.77296 (12)	0.0495 (6)
O6	0.5317 (3)	0.96367 (14)	0.88673 (11)	0.0548 (5)
O7	0.4993 (3)	1.09272 (18)	0.94041 (11)	0.0682 (7)
C19	0.4573 (4)	0.7890 (2)	0.96537 (13)	0.0467 (7)
H19A	0.4264	0.7413	0.9967	0.056*
H19B	0.4997	0.8448	0.9876	0.056*
N2	−0.1735 (3)	0.82995 (16)	0.79151 (10)	0.0389 (6)
C9	−0.2765 (4)	0.9463 (2)	0.71762 (13)	0.0469 (7)
H9A	−0.2093	0.9333	0.6817	0.056*
H9B	−0.3724	0.9751	0.7022	0.056*
N4	0.5926 (3)	1.0332 (2)	0.91780 (13)	0.0536 (7)
C5	0.2324 (4)	0.64897 (19)	0.92502 (13)	0.0462 (7)
H5	0.3154	0.6266	0.9492	0.055*

C20	0.5803 (4)	0.7464 (2)	0.92445 (16)	0.0607 (9)
H20A	0.6188	0.7953	0.8960	0.091*
H20B	0.5374	0.6935	0.9006	0.091*
H20C	0.6642	0.7230	0.9503	0.091*
O11	0.0986 (3)	0.99550 (19)	1.08050 (11)	0.0783 (8)
O8	0.7340 (3)	1.0388 (2)	0.92367 (13)	0.0916 (9)
O4	0.3972 (3)	1.0700 (2)	0.71906 (11)	0.0833 (9)
C8	−0.3115 (4)	0.8526 (2)	0.75264 (14)	0.0473 (7)
H8A	−0.4025	0.8606	0.7793	0.057*
H8B	−0.3314	0.8004	0.7229	0.057*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Y1	0.03256 (14)	0.02959 (11)	0.03916 (12)	−0.00066 (13)	−0.00454 (10)	−0.00200 (11)
Ni1	0.03277 (19)	0.02569 (14)	0.04342 (17)	−0.00143 (14)	−0.01103 (16)	−0.00396 (14)
O1	0.0352 (10)	0.0254 (8)	0.0483 (10)	0.0021 (8)	−0.0072 (8)	−0.0038 (8)
C10	0.0387 (18)	0.0466 (16)	0.0410 (15)	0.0092 (14)	−0.0048 (12)	−0.0015 (12)
C16	0.0349 (16)	0.0253 (12)	0.0377 (13)	0.0029 (10)	0.0047 (11)	−0.0004 (10)
O13	0.0424 (12)	0.0289 (9)	0.0456 (11)	−0.0013 (8)	−0.0055 (9)	−0.0074 (8)
C15	0.0331 (15)	0.0291 (12)	0.0402 (14)	−0.0009 (11)	0.0026 (12)	0.0019 (10)
C2	0.0400 (17)	0.0309 (12)	0.0433 (15)	−0.0048 (12)	0.0069 (13)	−0.0064 (12)
C11	0.0334 (16)	0.0357 (13)	0.0429 (16)	0.0025 (12)	−0.0032 (12)	−0.0005 (12)
C7	0.0417 (19)	0.0394 (15)	0.0494 (18)	−0.0107 (13)	0.0030 (14)	−0.0148 (13)
C1	0.0405 (17)	0.0259 (12)	0.0383 (14)	−0.0012 (11)	0.0039 (12)	−0.0013 (10)
C6	0.0403 (17)	0.0294 (12)	0.0390 (14)	0.0021 (11)	0.0051 (12)	0.0000 (10)
C17	0.055 (2)	0.0381 (14)	0.0409 (15)	−0.0038 (15)	−0.0079 (14)	−0.0088 (11)
C4	0.071 (2)	0.0272 (13)	0.0594 (19)	0.0013 (13)	0.0117 (17)	0.0040 (13)
C3	0.064 (2)	0.0268 (13)	0.0552 (18)	−0.0091 (14)	0.0070 (15)	−0.0060 (12)
C18	0.057 (2)	0.0402 (16)	0.077 (2)	−0.0083 (15)	−0.0175 (18)	0.0010 (16)
C13	0.047 (2)	0.0308 (14)	0.075 (2)	0.0094 (14)	0.0026 (16)	−0.0015 (14)
C12	0.0432 (19)	0.0398 (14)	0.062 (2)	0.0097 (13)	−0.0072 (15)	0.0051 (14)
C14	0.0482 (19)	0.0293 (13)	0.0551 (17)	−0.0003 (12)	0.0054 (14)	−0.0092 (12)
O9	0.0576 (15)	0.0562 (12)	0.0505 (11)	−0.0074 (12)	−0.0091 (10)	−0.0025 (10)
O5	0.0527 (14)	0.0427 (10)	0.0541 (12)	0.0036 (9)	0.0056 (11)	0.0048 (11)
O2	0.0421 (13)	0.0247 (8)	0.0565 (12)	−0.0014 (8)	−0.0142 (9)	−0.0018 (8)
N1	0.0351 (13)	0.0417 (13)	0.0437 (13)	0.0018 (11)	−0.0075 (9)	−0.0044 (11)
O10	0.0507 (13)	0.0757 (16)	0.0522 (12)	−0.0048 (12)	−0.0020 (11)	0.0029 (10)
O12	0.0376 (12)	0.0344 (10)	0.0503 (12)	0.0038 (8)	−0.0097 (9)	0.0000 (8)
N5	0.0618 (18)	0.0365 (12)	0.0484 (15)	−0.0012 (14)	0.0047 (12)	0.0043 (12)
O3	0.0576 (15)	0.0544 (13)	0.0491 (12)	0.0010 (11)	−0.0020 (10)	−0.0117 (10)
N3	0.0395 (15)	0.0683 (17)	0.0408 (15)	0.0204 (14)	−0.0014 (11)	0.0085 (14)
O6	0.0433 (13)	0.0440 (11)	0.0772 (14)	−0.0010 (10)	−0.0049 (11)	−0.0021 (10)
O7	0.0563 (17)	0.0776 (17)	0.0709 (16)	−0.0113 (13)	−0.0022 (12)	−0.0279 (13)
C19	0.0417 (18)	0.0483 (16)	0.0501 (17)	0.0057 (15)	−0.0081 (14)	0.0042 (13)
N2	0.0347 (14)	0.0368 (12)	0.0451 (13)	−0.0033 (11)	−0.0018 (10)	−0.0098 (10)
C9	0.0425 (18)	0.0510 (15)	0.0473 (15)	0.0054 (15)	−0.0115 (13)	−0.0132 (13)
N4	0.0434 (17)	0.0622 (17)	0.0552 (15)	−0.0102 (13)	−0.0036 (13)	0.0076 (13)

C5	0.054 (2)	0.0352 (14)	0.0495 (16)	0.0057 (14)	0.0043 (15)	0.0059 (12)
C20	0.048 (2)	0.055 (2)	0.079 (2)	0.0133 (17)	0.0066 (17)	0.0102 (16)
O11	0.112 (2)	0.0727 (15)	0.0500 (13)	−0.0111 (17)	0.0180 (13)	0.0028 (13)
O8	0.0366 (15)	0.134 (2)	0.1042 (19)	−0.0233 (16)	−0.0113 (13)	0.0043 (18)
O4	0.083 (2)	0.120 (2)	0.0471 (13)	0.0460 (18)	0.0175 (12)	0.0218 (14)
C8	0.0385 (18)	0.0504 (17)	0.0531 (18)	−0.0053 (14)	−0.0102 (14)	−0.0131 (14)

Geometric parameters (Å, °)

Y1—O1	2.3073 (17)	C4—C3	1.356 (4)
Y1—O2	2.3602 (18)	C4—C5	1.392 (4)
Y1—O3	2.472 (2)	C4—H4	0.9300
Y1—O5	2.451 (2)	C3—H3	0.9300
Y1—O6	2.409 (2)	C18—H18A	0.9600
Y1—O7	2.560 (2)	C18—H18B	0.9600
Y1—O9	2.399 (2)	C18—H18C	0.9600
Y1—O10	2.454 (2)	C13—C12	1.364 (4)
Y1—O12	2.6424 (17)	C13—C14	1.392 (4)
Y1—O13	2.5708 (17)	C13—H13	0.9300
Ni1—O1	1.8938 (17)	C12—H12	0.9300
Ni1—O2	1.8927 (18)	C14—H14	0.9300
Ni1—N1	1.907 (2)	O9—N5	1.274 (3)
Ni1—N2	1.908 (2)	O5—N3	1.276 (3)
O1—C16	1.333 (3)	N1—C9	1.475 (3)
C10—N1	1.286 (3)	O10—N5	1.262 (3)
C10—C11	1.447 (4)	O12—C19	1.450 (3)
C10—H10	0.9300	N5—O11	1.211 (3)
C16—C15	1.395 (4)	O3—N3	1.250 (3)
C16—C11	1.410 (4)	N3—O4	1.227 (3)
O13—C15	1.390 (3)	O6—N4	1.272 (3)
O13—C17	1.459 (3)	O7—N4	1.242 (3)
C15—C14	1.379 (3)	C19—C20	1.486 (4)
C2—C1	1.412 (4)	C19—H19A	0.9700
C2—C3	1.414 (4)	C19—H19B	0.9700
C2—C7	1.436 (4)	N2—C8	1.475 (4)
C11—C12	1.408 (4)	C9—C8	1.517 (4)
C7—N2	1.284 (3)	C9—H9A	0.9700
C7—H7	0.9300	C9—H9B	0.9700
C1—O2	1.333 (3)	N4—O8	1.223 (3)
C1—C6	1.392 (4)	C5—H5	0.9300
C6—C5	1.386 (3)	C20—H20A	0.9600
C6—O12	1.387 (3)	C20—H20B	0.9600
C17—C18	1.504 (4)	C20—H20C	0.9600
C17—H17A	0.9700	C8—H8A	0.9700
C17—H17B	0.9700	C8—H8B	0.9700
O1—Y1—O2	65.69 (6)	O13—C17—C18	112.2 (2)
O1—Y1—O3	75.50 (7)	O13—C17—H17A	109.2
O1—Y1—O5	74.16 (7)	C18—C17—H17A	109.2
O1—Y1—O6	142.25 (7)	O13—C17—H17B	109.2

O1—Y1—O7	130.85 (8)	C18—C17—H17B	109.2
O1—Y1—O9	124.85 (7)	H17A—C17—H17B	107.9
O1—Y1—O10	80.25 (7)	C3—C4—C5	120.7 (3)
O1—Y1—O12	126.25 (6)	C3—C4—H4	119.6
O1—Y1—O13	62.86 (6)	C5—C4—H4	119.6
O2—Y1—O3	69.65 (7)	C4—C3—C2	121.3 (3)
O2—Y1—O5	115.01 (6)	C4—C3—H3	119.3
O2—Y1—O6	113.00 (7)	C2—C3—H3	119.3
O2—Y1—O7	162.14 (7)	C17—C18—H18A	109.5
O2—Y1—O9	114.04 (7)	C17—C18—H18B	109.5
O2—Y1—O10	72.17 (7)	H18A—C18—H18B	109.5
O2—Y1—O12	60.71 (6)	C17—C18—H18C	109.5
O2—Y1—O13	123.36 (6)	H18A—C18—H18C	109.5
O3—Y1—O7	105.42 (8)	H18B—C18—H18C	109.5
O3—Y1—O12	89.47 (7)	C12—C13—C14	120.1 (3)
O3—Y1—O13	116.54 (7)	C12—C13—H13	119.9
O5—Y1—O3	51.71 (7)	C14—C13—H13	119.9
O5—Y1—O7	69.98 (8)	C13—C12—C11	121.5 (3)
O5—Y1—O10	146.17 (7)	C13—C12—H12	119.2
O5—Y1—O12	132.95 (6)	C11—C12—H12	119.2
O5—Y1—O13	71.44 (6)	C15—C14—C13	120.0 (3)
O6—Y1—O3	69.61 (8)	C15—C14—H14	120.0
O6—Y1—O5	73.16 (7)	C13—C14—H14	120.0
O6—Y1—O7	50.55 (7)	N5—O9—Y1	97.60 (16)
O6—Y1—O10	136.94 (8)	N3—O5—Y1	96.04 (16)
O6—Y1—O12	68.19 (6)	C1—O2—Ni1	125.48 (18)
O6—Y1—O13	121.83 (7)	C1—O2—Y1	130.33 (17)
O7—Y1—O12	102.84 (7)	Ni1—O2—Y1	104.11 (7)
O7—Y1—O13	74.42 (7)	C10—N1—C9	123.6 (2)
O9—Y1—O3	159.49 (8)	C10—N1—Ni1	125.9 (2)
O9—Y1—O5	130.82 (8)	C9—N1—Ni1	110.50 (18)
O9—Y1—O6	91.16 (8)	N5—O10—Y1	95.28 (18)
O9—Y1—O7	64.52 (8)	C6—O12—C19	117.0 (2)
O9—Y1—O10	52.16 (7)	C6—O12—Y1	118.95 (15)
O9—Y1—O12	76.33 (7)	C19—O12—Y1	123.98 (16)
O9—Y1—O13	79.21 (7)	O11—N5—O10	123.1 (3)
O10—Y1—O3	140.57 (8)	O11—N5—O9	122.2 (3)
O10—Y1—O7	113.97 (7)	O10—N5—O9	114.6 (2)
O10—Y1—O12	80.31 (7)	N3—O3—Y1	95.79 (16)
O10—Y1—O13	77.38 (7)	O4—N3—O3	123.0 (3)
O13—Y1—O12	153.82 (7)	O4—N3—O5	120.6 (3)
O1—Ni1—N1	94.30 (8)	O3—N3—O5	116.4 (2)
O1—Ni1—N2	177.80 (9)	N4—O6—Y1	100.24 (18)
O2—Ni1—O1	83.93 (7)	N4—O7—Y1	93.76 (17)
O2—Ni1—N1	172.48 (10)	O12—C19—C20	112.7 (2)
O2—Ni1—N2	95.83 (9)	O12—C19—H19A	109.1
N1—Ni1—N2	86.22 (10)	C20—C19—H19A	109.1
C16—O1—Ni1	123.80 (16)	O12—C19—H19B	109.1
C16—O1—Y1	128.40 (16)	C20—C19—H19B	109.1

Ni1—O1—Y1	106.08 (7)	H19A—C19—H19B	107.8
N1—C10—C11	123.8 (3)	C7—N2—C8	122.2 (2)
N1—C10—H10	118.1	C7—N2—Ni1	124.9 (2)
C11—C10—H10	118.1	C8—N2—Ni1	112.67 (17)
O1—C16—C15	116.8 (2)	N1—C9—C8	108.1 (2)
O1—C16—C11	123.2 (2)	N1—C9—H9A	110.1
C15—C16—C11	120.0 (2)	C8—C9—H9A	110.1
C15—O13—C17	117.79 (19)	N1—C9—H9B	110.1
C15—O13—Y1	117.82 (13)	C8—C9—H9B	110.1
C17—O13—Y1	124.28 (16)	H9A—C9—H9B	108.4
C14—C15—O13	125.9 (2)	O8—N4—O7	124.0 (3)
C14—C15—C16	120.5 (3)	O8—N4—O6	120.6 (3)
O13—C15—C16	113.5 (2)	O7—N4—O6	115.4 (3)
C1—C2—C3	118.1 (3)	C6—C5—C4	119.2 (3)
C1—C2—C7	123.8 (2)	C6—C5—H5	120.4
C3—C2—C7	118.1 (3)	C4—C5—H5	120.4
C12—C11—C16	117.9 (2)	C19—C20—H20A	109.5
C12—C11—C10	118.1 (3)	C19—C20—H20B	109.5
C16—C11—C10	124.0 (2)	H20A—C20—H20B	109.5
N2—C7—C2	125.8 (3)	C19—C20—H20C	109.5
N2—C7—H7	117.1	H20A—C20—H20C	109.5
C2—C7—H7	117.1	H20B—C20—H20C	109.5
O2—C1—C6	116.6 (2)	N2—C8—C9	107.0 (2)
O2—C1—C2	123.9 (3)	N2—C8—H8A	110.3
C6—C1—C2	119.4 (2)	C9—C8—H8A	110.3
C5—C6—O12	125.5 (3)	N2—C8—H8B	110.3
C5—C6—C1	121.1 (3)	C9—C8—H8B	110.3
O12—C6—C1	113.3 (2)	H8A—C8—H8B	108.6

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C20—H20A···O6	0.96	2.44	3.121 (4)	128
C9—H9B···O4^{i}	0.97	2.40	3.278 (4)	151
C7—H7···O4^{ii}	0.93	2.38	3.293 (4)	167

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

Fig. 1

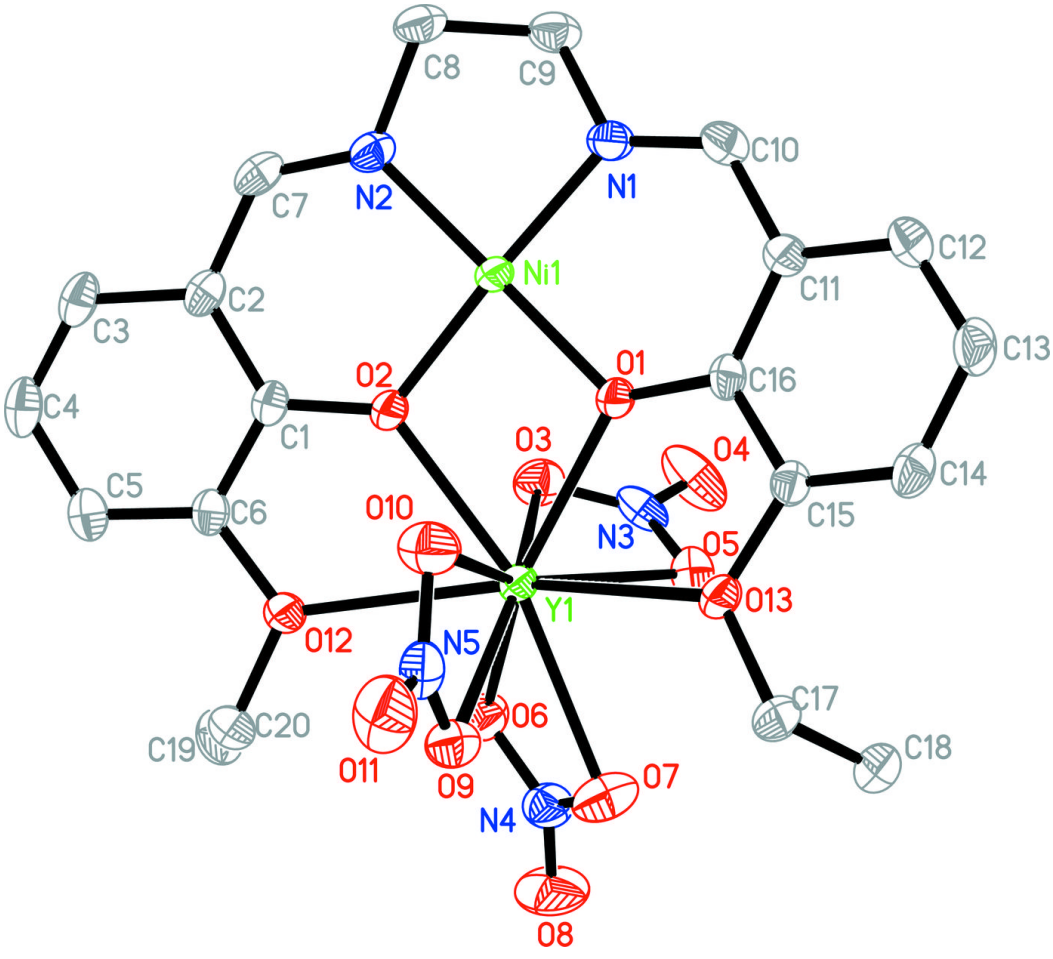

C8
C9
C10
N2
N1
C7
C12
Ni1
C11
C2
C3
C13
O2
O1
C16
C4
C1
O3
O4
C14
C5
C6
O10
N3
C15
O5
Y1
O13
O12
N5
C17
C19
C20
O6
C18
O11
O9
O7
N4
O8

Fig. 2