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Retraction of articles

This article reports the retraction of 39 articles published in *Acta Crystallographica Section E* between 2004 and 2009.

After thorough investigation (see Harrison *et al.*, 2010), 39 additional articles are retracted as a result of problems with the data sets or incorrect atom assignments. Full details of all the articles are given in Table 1.

Table 1
Details of articles to be retracted, in order of publication.

Title	Reference	Retracted by	DOI	Refcode
trans-Bis{1-[3-(cyclohexylamino)propyliminomethyl]-2-naphtholato}copper(II) dichloride dihydrate	Zhang (2004)	Journal	10.1107/S1600536804028296	BIPDUA
Bis(4-bromo-2-formylphenolato-κ^2O,O')copper(II)	Sun & Gao (2005)	Author	10.1107/S160053680500187X	FEYSUY
Bis(salicylaldehydo)zinc(II)	Xiong & Liu (2005)	Journal	10.1107/S1600536805010913	GAMDUU
Bis(4-bromo-2-formylphenolato-κ^2O,O')zinc(II)	Chen (2006)	Journal	10.1107/S1600536805040432	SAZCUS
Bis(2-formylphenolato-κ^2O,O')nickel(II)	Li & Chen (2006)	Journal	10.1107/S1600536806012931	IDAZAP
Bis(2-formylphenolato)cobalt(II)	Qiu (2006)	Journal	10.1107/S1600536806015704	GEJDUV
Bis(2-formylphenolato-κ^2O,O')manganese(II)	Wang & Fang (2006)	Journal	10.1107/S1600536806021039	IDOVED
Tetraaqua(1,10-phenanthroline-κ^2N,N')copper(II) naphthalene-1,5-disulfonate dihydrate	Liu *et al.* (2006)	Author	10.1107/S1600536806030637	GENYOO
Tetraaqua(1,10-phenanthroline-κ^2N,N')nickel(II) naphthalene-1,5-disulfonate dihydrate	Liu & Fan (2006)	Author	10.1107/S1600536806035410	KERBEP
{6,6′-Diethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-trinitratolutetium(III)copper(II)	Sui *et al.* (2006)	Journal	10.1107/S160053680604565X	HESPEB
Bis(2-formylphenolato-κ^2O,O')iron(II)	Yang *et al.* (2007)	Author	10.1107/S1600536807021721	PIFCAJ
2,6-Dimethoxybenzohydrazide	Qadeer *et al.* (2007*a*)	Journal	10.1107/S1600536807022593	PIFHES
2-(2,4-Dichlorophenylsulfanyl)acetohydrazide	Qadeer *et al.* (2007*b*)	Journal	10.1107/S1600536807022891	YIFSOW
{6,6′-Diethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-trinitratoeuropium(III)zinc(II)	Hu *et al.* (2007)	Author	10.1107/S1600536807031121	WIHKEE
{μ-6,6′-Diethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-trinitratocerium(III)zinc(II)	Sui, Zhang, Hu & Yin (2007)	Author	10.1107/S1600536807032564	WIHREL
{μ-6,6′-Diethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-trinitratopraseodymium(III)zinc(II)	Chen *et al.* (2007)	Author	10.1107/S1600536807032540	WIHRIP
{μ-6,6′-Diethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-trinitratopraseodymium(III)nickel(II)	Sui, Li *et al.* (2007)	Author	10.1107/S1600536807032618	UFACUA
{6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato-1$\kappa^4O^1,O^{1'},O^6,O^{6'}$:2$\kappa^4O^1,N,N',O^{1'}$}(methanol-1κO)-μ-nitrato-1:2κ^2O:O′-dinitrato-1κ^4O,O'-cerium(III)zinc(II)	Sui, Fang, Hu & Lin (2007)	Author	10.1107/S1600536807033314	UDUYIC
{6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-methanol-μ-nitrato-dinitratosamarium(III)nickel(II)	Sui, Zhang, Hu & Jiang (2007)	Author	10.1107/S1600536807037130	AFECEU
{6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-methanol-μ-nitrato-dinitratopraseodymium(III)zinc(II)	Sui, Fang & Yuan (2007)	Author	10.1107/S1600536807037488	AFICEY
{6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-methanol-μ-nitrato-dinitratolutetium(III)zinc(II)	Sui, Sui *et al.* (2007)	Author	10.1107/S1600536807037737	AFEFOH
catena-Poly[[chloridonickel(II)]-di-μ-chlorido-[chloridonickel(II)]-μ-4,4′-methylenebis(3,5-dimethylpyrazole)-κ^2N^2:$N^{2'}$]	Huang & Chen (2007)	Author	10.1107/S1600536807039384	VIJYOD
{2,2′-[o-Phenylenebis(nitrilomethylidyne)]diphenolato}zinc(II)	Liu *et al.* (2007*a*)	Author	10.1107/S1600536807040640	DIKYUS
trans-Bis(ethylenediamine-κ^2N,N')bis(nitrato-κO)zinc(II)	Liu, Zeng & Chen (2007)	Author	10.1107/S1600536807042390	XIKYEW
[N,N′-(o-Phenylene)bis(picolinamido)-κ^4N,N',N'',N''']cobalt(II)	Liu & Zeng (2007*a*)	Author	10.1107/S1600536807044571	XILFII
[N,N′-(o-Phenylene)dipicolinamide-κ^4N]nickel(II)	Liu & Zeng (2007*b*)	Author	10.1107/S1600536807048386	WINWEW
{2,2′-[o-Phenylenebis(nitrilomethylidyne)]diphenolato}manganese(II)	Liu *et al.* (2007*b*)	Author	10.1107/S1600536807052993	VIQPIV
N-(2-Amino-3-pyridyl)urea monohydrate	Li *et al.* (2007)	Author	10.1107/S1600536807047526	SIMFEA
N-(2-Fluorophenyl)carbamic acid monohydrate	Yang (2007)	Author	10.1107/S1600536807052464	WINMOW
Aqua(dimethylglyoxime-κ^2N,N')(3,5-dinitro-2-oxidobenzoato-κ^2O^1,O^2)-copper(II)	Liu & Wen (2007)	Author	10.1107/S1600536807054244	HIQCAM
μ-Acetato-tri-μ-ferrocenecarboxylatobis[(N,N-dimethylformamide)-copper(II)]	Liu, Lin *et al.* (2007)	Journal	10.1107/S1600536807059041	HIQQEE

 doi:10.1107/S1600536809054300

Table 1 (continued)

Title	Reference	Retracted by	DOI	Refcode
{μ-6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-μ-nitrato-dinitratoeuropium(III)zinc(II)	Hu *et al.* (2008)	Author	10.1107/S160053680706151X	MIRPAF
Bis(4-chloro-2-formylphenolato)nickel(II)	Li *et al.* (2008)	Author	10.1107/S1600536807056309	RISTET
{μ-6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-μ-nitrato-dinitratoterbium(III)zinc(II)	Chen *et al.* (2008)	Author	10.1107/S1600536808006958	QIXHIP
Bis(2-ethoxy-6-formylphenolato-κ^2O^1,O^6)nickel(II)	Han (2008)	Journal	10.1107/S160053680800809X	QIXLIT
{μ-6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-μ-nitrato-dinitratoholmium(III)zinc(II)	Xiao, Sui *et al.* (2008)	Author	10.1107/S1600536808013743	BIZTUA
{μ-6,6′-Diethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato}-trinitratoholmium(III)nickel(II)	Xiao, Fu *et al.* (2008)	Author	10.1107/S1600536808013755	BIZVAI
Hydrogen-bonding patterns in the cocrystal terephthalic acid–4,4′-bipyridine (2/1)	Wang *et al.* (2009)	Journal	10.1107/S160053680903236X	DUCZEH
{6,6′-Dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato-1$\kappa^4O^1,O^{1'},O^6,O^{6'}$:2$\kappa^4O^1,N,N',O^{1'}$}(ethanol-1κO)-μ-nitrato-1:2κ^2O:O′-dinitrato-1κ^4O,O′-samarium(III)zinc(II)	Huang *et al.* (2009)	Journal	10.1107/S1600536809033558	YUCWAV

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

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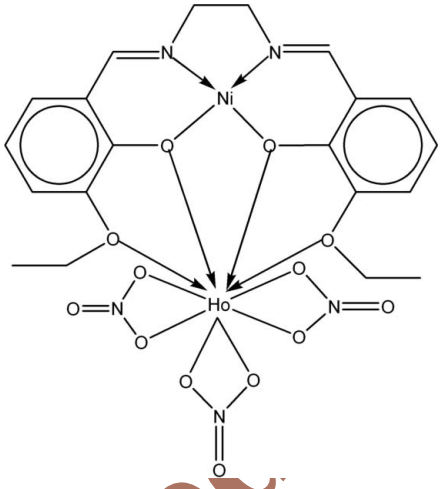
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.010 Å; R factor = 0.043; wR factor = 0.114; data-to-parameter ratio = 16.4.

In the title heteronuclear Ni^{II}–Ho^{III} complex (systematic name: {μ-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$\kappa^6O,O'$-holmium(III)nickel(II)), $[HoNi(C_{20}H_{22}N_2O_4)(NO_3)_3]$, with the hexadentate Schiff base compartmental ligand N,N'-bis(3-ethoxysalicylidene)ethylenediamine (H_2L), the Ho and Ni atoms are doubly bridged by two phenolate O atoms of the Schiff base ligand. The coordination of Ni is square-planar with the donor centers of two imine N atoms and two phenolate O atoms. The holmium(III) center has a tenfold coordination environment of O atoms, involving the phenolate O atoms, two ethoxy O atoms and two O atoms each from the three nitrates. Weak C—H···O and O···Ni [3.383 (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

$[HoNi(C_{20}H_{22}N_2O_4)(NO_3)_3]$
M_r = 764.07
Orthorhombic, $P2_12_12_1$
a = 8.5825 (8) Å
b = 13.7028 (14) Å
c = 21.203 (2) Å
V = 2493.6 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.98 mm^{-1}
T = 293 (2) K
0.17 × 0.16 × 0.13 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.559, T_{max} = 0.625
18705 measured reflections
5970 independent reflections
4299 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.113
S = 1.04
5970 reflections
364 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 2.00 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}
Absolute structure: Flack (1983), 2455 Friedel pairs
Flack parameter: −0.003 (18)

Table 1
Selected bond lengths (Å).

Ho1—O1	2.390 (4)	Ho1—O9	2.492 (5)
Ho1—O2	2.343 (4)	Ho1—O11	2.531 (5)
Ho1—O3	2.540 (4)	Ho1—O12	2.425 (5)
Ho1—O4	2.594 (4)	Ni1—O1	1.846 (4)
Ho1—O5	2.430 (5)	Ni1—O2	1.847 (4)
Ho1—O6	2.468 (5)	Ni1—N1	1.837 (5)
Ho1—O8	2.460 (5)	Ni1—N2	1.837 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12···O10^{i}	0.93	2.37	3.286 (8)	169
C10—H10A···O10^{ii}	0.97	2.42	3.301 (9)	150

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *APEX2*; software used to prepare material for publication: *APEX2* and *publCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2401).

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supporting information

Acta Cryst. (2008). E**64**, m806–m807 [doi:10.1107/S1600536808013755]

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

Yi-An Xiao, Xiang-Kai Fu, Yan Sui, Qing Wu and Shao-Hui Xiong

S1. 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}—Ho^{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 holmium 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 holmium(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 Ho1/O1/O2 is 6.97 (26)° suggesting that the bridging moiety is almost planar; the deviation of atoms from the least squares Ni1/O1/O2/Ho1 plane being -0.0583 (2)Å for Ni, -0.0397 (3)Å for Ho, 0.0483 (2)Å for O1 and 0.0497 (2)Å for O2.

The holmium(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 holmium to complete the decacoordination. The three kinds of Ho—O bond distances are significantly different, the shortest being the Ho—O(phenolate) and longest being the Ho—*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 0.0698 (2) Å, while Ni1 is 0.0022 (2)Å above this square plane.

Adjacent molecules are held together by weak interactions (O13···Ni1=3.383 (4) Å, C10—H10A···$O10^{i}$=3.301 (9), and C12—H12···$O10^{ii}$=3.286 (8); symmetry codes:(i)*x* - 1, *y*, *z*; (ii)-*x*, 1/2 + *y*, 1/2 - *z*.) these link the molecules into a two-dimensional zigzag sheet(Fig 2).

S2. 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 holmium(III) nitrate hexahydrate (0.459 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 $C_{20}H_{22}HoN_5NiO_{13}$: C 31.44, H 2.90, Ho 21.59, N 9.17, Ni 7.68%; found: C 31.85, H 2.95, Ho 21.55, N 9.24, Ni 7.78. IR(KBr, cm^{-1}): 1645(C=N), 1385,1491(nitrate).

S3. 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.

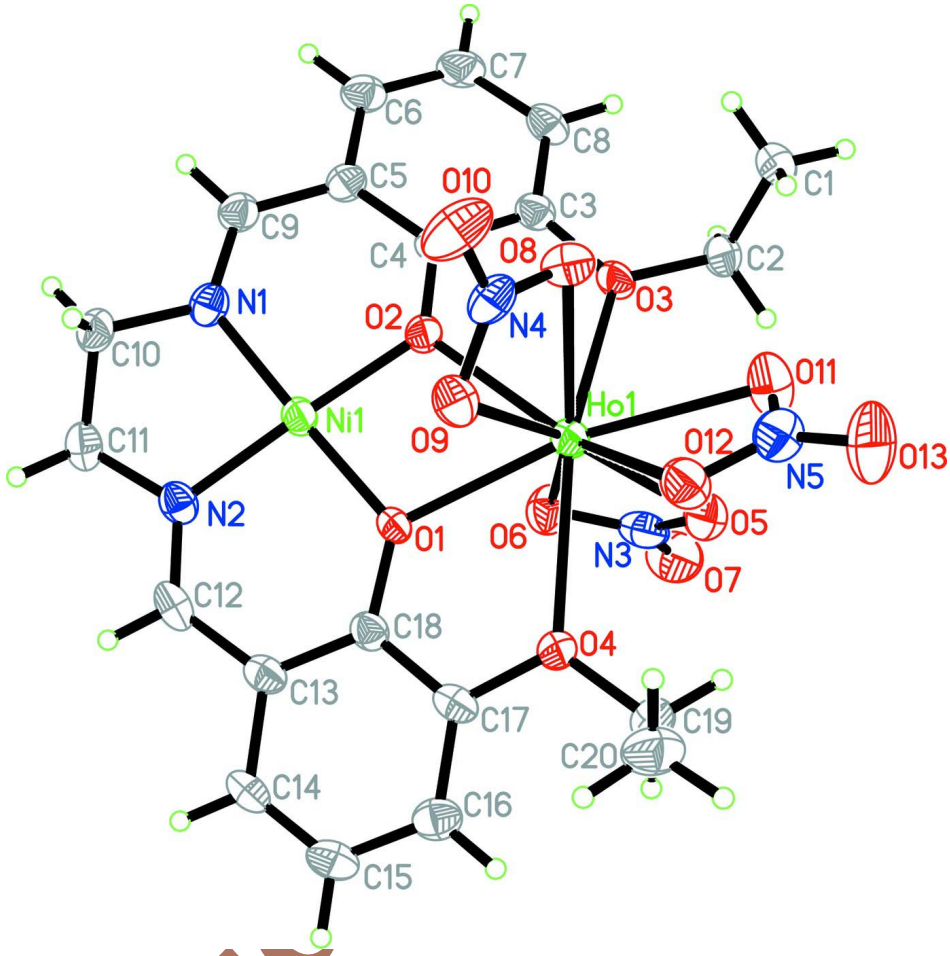

Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids.

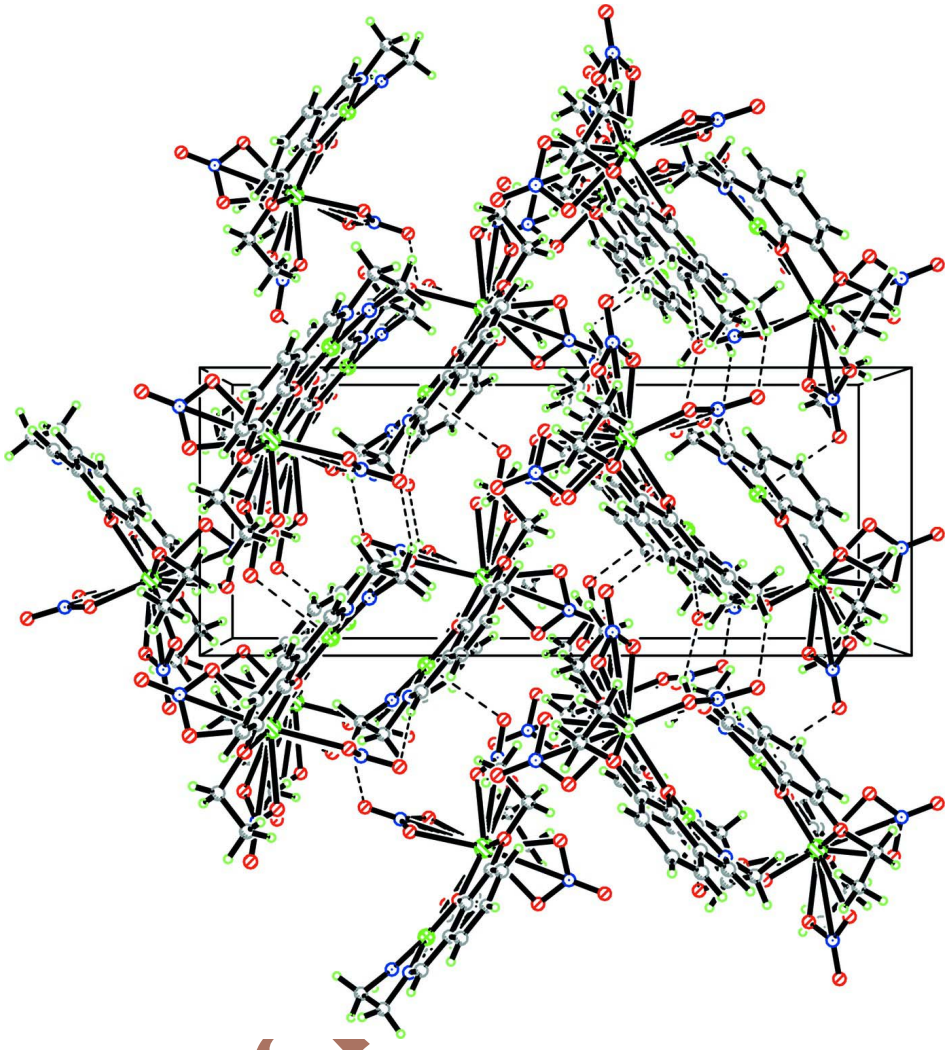

Figure 2

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

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

Crystal data

[HoNi($C_{20}H_{22}N_2O_4$)(NO_3)$_3$]
M_r = 764.07
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
a = 8.5825 (8) Å
b = 13.7028 (14) Å
c = 21.203 (2) Å
V = 2493.6 (4) Å^3
Z = 4
F(000) = 1504
D_x = 2.035 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 6504 reflections
θ = 1.9–28.3°
μ = 3.98 mm^{-1}
T = 293 K
Block, red
0.17 × 0.16 × 0.13 mm

Data collection

Bruker APEXII area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.559, T_{max} = 0.626
18705 measured reflections
5970 independent reflections
4299 reflections with $I > 2\sigma(I)$
R_{int} = 0.055
θ_{max} = 28.3°, θ_{min} = 1.9°
h = −11→11
k = −17→18
l = −27→27

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.113
S = 1.04
5970 reflections
364 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 2.00 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0058 (4)
Absolute structure: Flack (1983), 2455 Friedel pairs
Absolute structure parameter: −0.003 (18)

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}
C5	−0.1509 (7)	0.3317 (5)	0.3044 (3)	0.0427 (15)
Ho1	0.25263 (3)	0.49982 (2)	0.400886 (12)	0.04143 (11)
Ni1	−0.06665 (9)	0.56000 (6)	0.31450 (4)	0.03838 (19)
O2	0.0323 (4)	0.4496 (3)	0.34434 (19)	0.0399 (10)
O1	0.0808 (5)	0.6237 (3)	0.36270 (19)	0.0411 (10)
N1	−0.1998 (6)	0.4934 (4)	0.2621 (2)	0.0434 (12)
N2	−0.1729 (6)	0.6703 (4)	0.2905 (3)	0.0423 (13)
N3	0.1352 (8)	0.5100 (5)	0.5279 (3)	0.0588 (16)
O6	0.0496 (6)	0.5277 (4)	0.4813 (2)	0.0647 (14)
C12	−0.1460 (7)	0.7577 (5)	0.3087 (3)	0.0470 (16)
H12	−0.2126	0.8065	0.2945	0.056*
O3	0.1551 (5)	0.3283 (3)	0.4234 (2)	0.0422 (10)
C18	0.0875 (7)	0.7189 (4)	0.3736 (3)	0.0386 (13)

O4	0.3135 (5)	0.6799 (3)	0.4295 (2)	0.0473 (11)
O5	0.2787 (6)	0.4948 (4)	0.5150 (2)	0.0609 (13)
C15	0.1149 (8)	0.9161 (5)	0.4034 (4)	0.0556 (18)
H15	0.1222	0.9816	0.4147	0.067*
C4	−0.0255 (7)	0.3594 (4)	0.3442 (3)	0.0387 (14)
O7	0.0834 (8)	0.5055 (4)	0.5815 (3)	0.0841 (17)
C19	0.4500 (8)	0.7097 (5)	0.4648 (3)	0.0533 (17)
H19A	0.4917	0.6538	0.4872	0.064*
H19B	0.4203	0.7583	0.4957	0.064*
C1	0.3381 (9)	0.1918 (6)	0.4400 (4)	0.059 (2)
H1A	0.2848	0.1544	0.4084	0.089*
H1B	0.3793	0.1487	0.4716	0.089*
H1C	0.4219	0.2275	0.4208	0.089*
C17	0.2135 (7)	0.7529 (4)	0.4098 (3)	0.0422 (15)
C13	−0.0195 (8)	0.7860 (4)	0.3498 (3)	0.0434 (15)
C14	−0.0046 (9)	0.8868 (5)	0.3667 (3)	0.0515 (17)
H14	−0.0773	0.9320	0.3524	0.062*
C2	0.2241 (8)	0.2632 (5)	0.4706 (3)	0.0487 (16)
H2A	0.1421	0.2270	0.4917	0.058*
H2B	0.2784	0.3017	0.5021	0.058*
O9	0.3184 (6)	0.5422 (4)	0.2897 (2)	0.0581 (13)
N4	0.3530 (6)	0.4577 (5)	0.2744 (3)	0.0517 (16)
O8	0.3482 (6)	0.3918 (3)	0.3167 (3)	0.0573 (12)
C9	−0.2254 (8)	0.3989 (5)	0.2623 (3)	0.0447 (15)
H9	−0.2964	0.3741	0.2333	0.054*
O12	0.5289 (6)	0.5328 (4)	0.3902 (3)	0.0593 (13)
N5	0.5831 (8)	0.4621 (5)	0.4212 (3)	0.0605 (16)
C16	0.2267 (9)	0.8516 (5)	0.4248 (3)	0.0510 (17)
H16	0.3099	0.8738	0.4489	0.061*
C6	−0.2073 (8)	0.2359 (5)	0.3070 (3)	0.0506 (17)
H6	−0.2863	0.2162	0.2798	0.061*
C3	0.0358 (7)	0.2907 (5)	0.3862 (3)	0.0417 (14)
C11	−0.3100 (8)	0.6507 (5)	0.2514 (3)	0.0516 (17)
H11A	−0.3272	0.7036	0.2218	0.062*
H11B	−0.4022	0.6434	0.2774	0.062*
C10	−0.2754 (8)	0.5578 (5)	0.2171 (3)	0.0513 (16)
H10A	−0.3710	0.5285	0.2016	0.062*
H10B	−0.2073	0.5702	0.1815	0.062*
O11	0.4839 (6)	0.4045 (4)	0.4421 (3)	0.0728 (17)
C20	0.5726 (10)	0.7506 (6)	0.4231 (4)	0.071 (2)
H20A	0.6009	0.7031	0.3919	0.106*
H20B	0.6624	0.7672	0.4478	0.106*
H20C	0.5336	0.8081	0.4026	0.106*
C8	−0.0229 (7)	0.1970 (5)	0.3887 (4)	0.0484 (16)
H8	0.0192	0.1514	0.4164	0.058*
O10	0.3939 (7)	0.4348 (5)	0.2204 (2)	0.0808 (18)
O13	0.7234 (6)	0.4536 (7)	0.4307 (3)	0.099 (2)
C7	−0.1465 (9)	0.1717 (5)	0.3492 (3)	0.0563 (19)

H7	−0.1884	0.1092	0.3518	0.068*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C5	0.040 (3)	0.041 (3)	0.048 (4)	−0.006 (3)	−0.003 (3)	−0.002 (3)
Ho1	0.04247 (16)	0.03543 (16)	0.04639 (17)	−0.00007 (18)	−0.00389 (12)	0.00217 (13)
Ni1	0.0375 (4)	0.0340 (4)	0.0436 (4)	−0.0005 (3)	−0.0050 (4)	0.0047 (4)
O2	0.038 (2)	0.033 (2)	0.049 (2)	−0.0065 (18)	−0.0099 (18)	0.004 (2)
O1	0.045 (2)	0.026 (2)	0.052 (2)	0.0024 (19)	−0.014 (2)	0.004 (2)
N1	0.037 (2)	0.051 (3)	0.043 (3)	−0.005 (3)	−0.002 (2)	0.002 (3)
N2	0.040 (3)	0.038 (3)	0.049 (3)	−0.001 (2)	0.001 (2)	0.010 (3)
N3	0.085 (5)	0.037 (3)	0.054 (3)	0.003 (3)	0.007 (3)	−0.005 (3)
O6	0.066 (3)	0.076 (4)	0.052 (3)	0.003 (3)	−0.001 (3)	−0.001 (3)
C12	0.041 (3)	0.046 (4)	0.054 (4)	0.011 (3)	0.009 (3)	0.018 (4)
O3	0.045 (2)	0.027 (2)	0.054 (3)	−0.0026 (19)	−0.004 (2)	0.008 (2)
C18	0.039 (3)	0.034 (3)	0.043 (3)	−0.002 (3)	0.000 (3)	0.006 (3)
O4	0.049 (2)	0.037 (2)	0.056 (3)	−0.004 (2)	−0.016 (2)	0.000 (2)
O5	0.070 (3)	0.061 (3)	0.051 (3)	0.005 (3)	−0.008 (2)	0.002 (3)
C15	0.063 (4)	0.034 (4)	0.069 (5)	−0.007 (3)	0.008 (4)	0.003 (4)
C4	0.041 (3)	0.036 (3)	0.039 (3)	−0.004 (3)	0.001 (3)	−0.001 (3)
O7	0.120 (5)	0.077 (4)	0.055 (3)	0.004 (4)	0.013 (4)	0.003 (3)
C19	0.048 (4)	0.052 (4)	0.060 (4)	−0.002 (3)	−0.010 (4)	−0.006 (3)
C1	0.057 (4)	0.044 (4)	0.077 (5)	0.010 (3)	−0.016 (4)	−0.002 (4)
C17	0.053 (4)	0.031 (3)	0.043 (3)	0.004 (3)	0.010 (3)	0.003 (3)
C13	0.052 (4)	0.032 (3)	0.046 (3)	0.001 (3)	0.005 (3)	0.004 (3)
C14	0.061 (4)	0.032 (3)	0.062 (4)	0.011 (3)	0.001 (3)	0.008 (3)
C2	0.061 (4)	0.042 (4)	0.043 (3)	0.002 (3)	−0.010 (3)	0.008 (3)
O9	0.069 (3)	0.051 (3)	0.055 (3)	0.002 (3)	−0.004 (3)	0.012 (3)
N4	0.043 (3)	0.063 (4)	0.049 (3)	−0.018 (3)	−0.006 (3)	−0.003 (3)
O8	0.064 (3)	0.051 (3)	0.056 (3)	−0.007 (2)	0.001 (3)	−0.009 (3)
C9	0.047 (4)	0.041 (3)	0.046 (3)	−0.006 (3)	−0.003 (3)	0.000 (3)
O12	0.053 (3)	0.052 (3)	0.073 (3)	0.000 (2)	0.000 (3)	0.005 (3)
N5	0.055 (4)	0.068 (4)	0.059 (3)	−0.002 (3)	0.002 (3)	0.000 (3)
C16	0.054 (4)	0.047 (4)	0.053 (3)	−0.011 (3)	0.005 (3)	−0.001 (3)
C6	0.047 (4)	0.041 (4)	0.064 (4)	−0.006 (3)	0.002 (4)	−0.007 (4)
C3	0.042 (3)	0.034 (3)	0.049 (3)	−0.001 (3)	0.001 (3)	0.001 (3)
C11	0.046 (4)	0.056 (5)	0.053 (4)	0.005 (3)	−0.008 (3)	0.011 (4)
C10	0.049 (4)	0.051 (4)	0.054 (3)	−0.007 (3)	−0.019 (3)	0.004 (3)
O11	0.054 (3)	0.081 (4)	0.083 (4)	0.005 (3)	−0.003 (3)	0.029 (3)
C20	0.066 (5)	0.058 (5)	0.087 (5)	−0.016 (5)	0.006 (5)	−0.012 (5)
C8	0.047 (4)	0.034 (4)	0.064 (4)	0.001 (3)	0.005 (3)	0.008 (3)
O10	0.080 (4)	0.115 (5)	0.048 (3)	−0.034 (4)	0.013 (3)	−0.018 (4)
O13	0.044 (3)	0.138 (6)	0.114 (5)	0.010 (4)	−0.012 (3)	0.022 (5)
C7	0.063 (4)	0.034 (4)	0.072 (5)	−0.013 (3)	0.000 (4)	−0.002 (4)

Geometric parameters (Å, º)

Ho1—O1	2.390 (4)	C15—H15	0.9300
Ho1—O2	2.343 (4)	C4—C3	1.398 (9)
Ho1—O3	2.540 (4)	C19—C20	1.484 (10)
Ho1—O4	2.594 (4)	C19—H19A	0.9700
Ho1—O5	2.430 (5)	C19—H19B	0.9700
Ho1—O6	2.468 (5)	C1—C2	1.529 (10)
Ho1—O8	2.460 (5)	C1—H1A	0.9600
Ho1—O9	2.492 (5)	C1—H1B	0.9600
Ho1—O11	2.531 (5)	C1—H1C	0.9600
Ho1—O12	2.425 (5)	C17—C16	1.394 (9)
Ni1—O1	1.846 (4)	C13—C14	1.433 (9)
Ni1—O2	1.847 (4)	C14—H14	0.9300
Ni1—N1	1.837 (5)	C2—H2A	0.9700
Ni1—N2	1.837 (5)	C2—H2B	0.9700
C5—C6	1.400 (9)	O9—N4	1.239 (8)
C5—C4	1.419 (8)	N4—O10	1.238 (7)
C5—C9	1.433 (9)	N4—O8	1.272 (8)
O2—C4	1.332 (7)	C9—H9	0.9300
O1—C18	1.327 (7)	O12—N5	1.259 (8)
N1—C9	1.313 (8)	N5—O13	1.227 (8)
N1—C10	1.453 (8)	N5—O11	1.242 (8)
N2—C12	1.280 (8)	C16—H16	0.9300
N2—C11	1.465 (9)	C6—C7	1.359 (10)
N3—O7	1.221 (7)	C6—H6	0.9300
N3—O6	1.255 (8)	C3—C8	1.380 (9)
N3—O5	1.279 (8)	C11—C10	1.496 (10)
C12—C13	1.445 (9)	C11—H11A	0.9700
C12—H12	0.9300	C11—H11B	0.9700
O3—C3	1.392 (7)	C10—H10A	0.9700
O3—C2	1.466 (7)	C10—H10B	0.9700
C18—C13	1.393 (8)	C20—H20A	0.9600
C18—C17	1.406 (9)	C20—H20B	0.9600
O4—C17	1.383 (7)	C20—H20C	0.9600
O4—C19	1.449 (8)	C8—C7	1.396 (10)
C15—C14	1.348 (10)	C8—H8	0.9300
C15—C16	1.382 (10)	C7—H7	0.9300

C6—C5—C4	119.3 (6)	C19—O4—Ho1	123.5 (4)
C6—C5—C9	118.2 (6)	N3—O5—Ho1	96.9 (4)
C4—C5—C9	122.4 (6)	C14—C15—C16	121.8 (6)
O2—Ho1—O1	62.44 (14)	C14—C15—H15	119.1
O2—Ho1—O12	142.78 (15)	C16—C15—H15	119.1
O1—Ho1—O12	116.07 (17)	O2—C4—C3	118.9 (5)
O2—Ho1—O5	125.12 (15)	O2—C4—C5	122.1 (6)
O1—Ho1—O5	114.48 (17)	C3—C4—C5	118.9 (5)
O12—Ho1—O5	90.49 (18)	O4—C19—C20	111.9 (6)

O2—Ho1—O8	73.78 (15)	O4—C19—H19A	109.2
O1—Ho1—O8	112.76 (15)	C20—C19—H19A	109.2
O12—Ho1—O8	73.62 (17)	O4—C19—H19B	109.2
O5—Ho1—O8	132.44 (19)	C20—C19—H19B	109.2
O2—Ho1—O6	80.15 (16)	H19A—C19—H19B	107.9
O1—Ho1—O6	71.83 (16)	C2—C1—H1A	109.5
O12—Ho1—O6	136.61 (17)	C2—C1—H1B	109.5
O5—Ho1—O6	51.82 (17)	H1A—C1—H1B	109.5
O8—Ho1—O6	146.11 (17)	C2—C1—H1C	109.5
O2—Ho1—O9	76.56 (16)	H1A—C1—H1C	109.5
O1—Ho1—O9	69.75 (16)	H1B—C1—H1C	109.5
O12—Ho1—O9	69.29 (18)	O4—C17—C16	125.6 (6)
O5—Ho1—O9	157.98 (18)	O4—C17—C18	113.8 (5)
O8—Ho1—O9	51.54 (17)	C16—C17—C18	120.6 (6)
O6—Ho1—O9	141.01 (17)	C18—C13—C14	119.1 (6)
O2—Ho1—O11	131.15 (18)	C18—C13—C12	122.4 (6)
O1—Ho1—O11	165.27 (17)	C14—C13—C12	118.5 (6)
O12—Ho1—O11	50.32 (17)	C15—C14—C13	119.9 (6)
O5—Ho1—O11	64.50 (19)	C15—C14—H14	120.0
O8—Ho1—O11	71.28 (19)	C13—C14—H14	120.0
O6—Ho1—O11	113.28 (17)	O3—C2—C1	111.0 (5)
O9—Ho1—O11	105.60 (18)	O3—C2—H2A	109.4
O2—Ho1—O3	63.79 (14)	C1—C2—H2A	109.4
O1—Ho1—O3	121.17 (14)	O3—C2—H2B	109.4
O12—Ho1—O3	120.80 (16)	C1—C2—H2B	109.4
O5—Ho1—O3	79.44 (17)	H2A—C2—H2B	108.0
O8—Ho1—O3	71.93 (16)	N4—O9—Ho1	94.8 (4)
O6—Ho1—O3	77.33 (17)	O10—N4—O9	123.1 (7)
O9—Ho1—O3	117.91 (17)	O10—N4—O8	118.8 (7)
O11—Ho1—O3	73.51 (16)	O9—N4—O8	118.1 (6)
O2—Ho1—O4	124.19 (15)	N4—O8—Ho1	95.5 (4)
O1—Ho1—O4	61.83 (14)	N1—C9—C5	124.2 (6)
O12—Ho1—O4	69.37 (16)	N1—C9—H9	117.9
O5—Ho1—O4	77.05 (16)	C5—C9—H9	117.9
O8—Ho1—O4	132.43 (16)	N5—O12—Ho1	99.7 (4)
O6—Ho1—O4	80.41 (17)	O13—N5—O11	123.6 (8)
O9—Ho1—O4	87.35 (17)	O13—N5—O12	121.4 (7)
O11—Ho1—O4	104.59 (17)	O11—N5—O12	114.9 (6)
O3—Ho1—O4	154.49 (18)	C15—C16—C17	119.3 (7)
N1—Ni1—N2	86.1 (3)	C15—C16—H16	120.3
N1—Ni1—O1	175.1 (2)	C17—C16—H16	120.3
N2—Ni1—O1	96.0 (2)	C7—C6—C5	120.0 (6)
N1—Ni1—O2	94.9 (2)	C7—C6—H6	120.0
N2—Ni1—O2	175.7 (2)	C5—C6—H6	120.0
O1—Ni1—O2	83.27 (17)	C8—C3—O3	126.2 (6)
C4—O2—Ni1	126.1 (4)	C8—C3—C4	120.9 (6)
C4—O2—Ho1	125.1 (4)	O3—C3—C4	112.9 (5)
Ni1—O2—Ho1	107.79 (18)	N2—C11—C10	105.8 (5)

C18—O1—Ni1	126.2 (4)	N2—C11—H11A	110.6
C18—O1—Ho1	127.8 (4)	C10—C11—H11A	110.6
Ni1—O1—Ho1	105.95 (17)	N2—C11—H11B	110.6
C9—N1—C10	121.8 (5)	C10—C11—H11B	110.6
C9—N1—Ni1	126.3 (5)	H11A—C11—H11B	108.7
C10—N1—Ni1	111.9 (4)	N1—C10—C11	106.7 (5)
C12—N2—C11	119.2 (6)	N1—C10—H10A	110.4
C12—N2—Ni1	126.7 (5)	C11—C10—H10A	110.4
C11—N2—Ni1	113.9 (4)	N1—C10—H10B	110.4
O7—N3—O6	122.0 (7)	C11—C10—H10B	110.4
O7—N3—O5	122.8 (7)	H10A—C10—H10B	108.6
O6—N3—O5	115.3 (6)	N5—O11—Ho1	95.0 (4)
N3—O6—Ho1	95.8 (4)	C19—C20—H20A	109.5
N2—C12—C13	124.7 (6)	C19—C20—H20B	109.5
N2—C12—H12	117.7	H20A—C20—H20B	109.5
C13—C12—H12	117.7	C19—C20—H20C	109.5
C3—O3—C2	117.3 (5)	H20A—C20—H20C	109.5
C3—O3—Ho1	118.6 (3)	H20B—C20—H20C	109.5
C2—O3—Ho1	124.0 (4)	C3—C8—C7	119.0 (6)
O1—C18—C13	123.9 (6)	C3—C8—H8	120.5
O1—C18—C17	117.0 (5)	C7—C8—H8	120.5
C13—C18—C17	119.1 (6)	C6—C7—C8	121.7 (6)
C17—O4—C19	117.0 (5)	C6—C7—H7	119.1
C17—O4—Ho1	119.5 (4)	C8—C7—H7	119.1

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C20—H20*A*···O12	0.96	2.41	3.088 (9)	127
C12—H12···O10^{i}	0.93	2.37	3.286 (8)	169
C10—H10*A*···O10^{ii}	0.97	2.42	3.301 (9)	150
C2—H2*B*···O11	0.97	2.59	3.015 (9)	107
C1—H1*C*···O11	0.96	2.52	3.173 (10)	125

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