

Poly[[[μ_3 -5-(pyridine-4-carboxamido)-isophthalato]{ μ_3 -5-[(pyridin-1-ium-4-yl)carbonylamino]isophthalato}-neodymium(III)] dihydrate]

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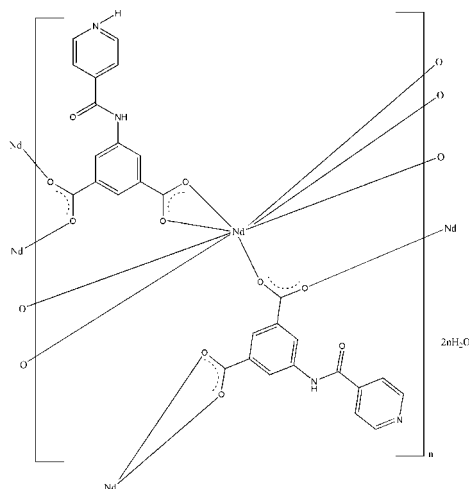
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 Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.038; wR factor = 0.101; data-to-parameter ratio = 13.0.

In the title compound, $[[\text{Nd}(\text{C}_{14}\text{H}_9\text{N}_2\text{O}_5)(\text{C}_{14}\text{H}_8\text{N}_2\text{O}_5)] \cdot 2\text{H}_2\text{O}]_n$, the Nd^{III} atom is eight-coordinated as it is surrounded by eight carboxylate O atoms from six ligands in a distorted square-antiprismatic arrangement. The Nd^{III} atoms are linked by HL^- and L^{2-} ligands [H_2L is 5-(pyridine-4-carboxamido)-isophthalic acid], forming a bilayer network. The layers are linked into a three-dimensional network through $\text{N}-\text{H} \cdots \text{O}$ and $\text{O}-\text{H} \cdots \text{O}$ hydrogen bonds.

Related literature

For background on transition metal complexes that exhibit one-, two- and three-dimensional frameworks, see: Kitagawa & Kondo (1998). For high-dimensional lanthanide frameworks, see: Kiritsis *et al.* (1998); Zhao *et al.* (2004). For coordination capabilities of carboxylate, pyridine and amide groups, see: Huyskens (1977); Lee & Kumler (1962); Wang *et al.* (2007).



Experimental

Crystal data

$[\text{Nd}(\text{C}_{14}\text{H}_9\text{N}_2\text{O}_5)(\text{C}_{14}\text{H}_8\text{N}_2\text{O}_5)] \cdot 2\text{H}_2\text{O}$
 $M_r = 749.73$
 Monoclinic, $P2_1/c$
 $a = 13.4421$ (15) Å
 $b = 13.7754$ (17) Å
 $c = 16.2418$ (13) Å
 $\beta = 115.813$ (4)°
 $V = 2707.4$ (5) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 2.00$ mm⁻¹
 $T = 291$ K
 $0.18 \times 0.16 \times 0.12$ mm

Data collection

Bruker SMART APEX diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 2008)
 $T_{\text{min}} = 0.715$, $T_{\text{max}} = 0.796$
 14297 measured reflections
 5287 independent reflections
 4792 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.052$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.101$
 $S = 1.07$
 5287 reflections
 406 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.83$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.61$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$\text{N2}-\text{H2} \cdots \text{O1W}$	0.86	2.06	2.846 (5)	151
$\text{N1}-\text{H1} \cdots \text{O2}^i$	0.89	1.85	2.725 (4)	171
$\text{O1W}-\text{H1WA} \cdots \text{O9}^j$	0.85	1.89	2.734 (5)	177
$\text{O2W}-\text{H2WA} \cdots \text{O10}^k$	0.85	2.52	3.124 (5)	129
$\text{O1W}-\text{H1WB} \cdots \text{O10}^{ii}$	0.85	2.00	2.845 (5)	171
$\text{O2W}-\text{H2WA} \cdots \text{O4}^{iii}$	0.85	2.36	2.923 (5)	125
$\text{O2W}-\text{H2WB} \cdots \text{O1}^{iv}$	0.85	2.11	2.957 (4)	178
$\text{N4}-\text{H4A} \cdots \text{O2W}^v$	0.86	2.15	2.953 (5)	156

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

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2003); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5214).

References

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supplementary materials

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Poly[[[μ_3 -5-(pyridine-4-carboxamido)isophthalato]{ μ_3 -5-[(pyridin-1-ium-4-yl)carbonylamino]isophthalato}neodymium(III)] dihydrate]

Y.-F. Deng

Comment

In recent years, there has been a great deal of interest in synthesizing transition metal complexes that exhibit one-, two- and three-dimensional frameworks (Kitagawa & Kondo, 1998). However, high-dimensional lanthanide frameworks are less common (Kiritsis *et al.*, 1998; Zhao *et al.*, 2004). On the other hand, it is well known that carboxylate and pyridine groups have good coordination capacities as well as the amide group, a group with two different types of hydrogen bonding sites: the $-NH$ moiety that acts as an electron acceptor and a $-C=O$ group that acts as an electron donor (Lee, & Kumler, 1962; Huyskens, 1977; Wang *et al.*, 2007). The study reports a new lanthanide(III) coordination polymer, $[Nd(HL)(L)]_n \cdot 2nH_2O$, (I), with H_2L and $Nd(NO_3)_3 \cdot 6H_2O$.

In the title compound, the central Nd^{III} ion is eight-coordinated by eight O atoms from six ligands, which gives a square antiprismatic geometry (Fig. 1). The carboxyl groups of the two unique L^{2-} (HL^-) ligands exhibit the same coordination modes: there is a monocarboxylate and a dicarboxylate, i.e., the monocarboxylate group coordinates to one Nd^{III} atom in $\mu_1-\eta^1:\eta^1$ -chelate mode and the other dicarboxylate connects two Nd^{III} atoms in a $\mu_2-\eta^1:\eta^1$ bridging mode. The pyridyl groups are free. Based on the coordination modes of the carboxylate groups of L^{2-} (HL^-), a bilayer network is formed (Fig. 2). Adjacent molecules are linked through $N-H\cdots O$ and $O-H\cdots O$ hydrogen bonds into a three-dimensional network.

Experimental

A mixture of 0.05 mmol $Nd(NO_3)_3 \cdot 6H_2O$ (21.5 mg, 0.05 mmol), H_2L (28.6 mg, 0.1 mmol), NaOH (6.0 mg, 0.15 mmol), MeOH (4 ml) and H_2O (6 ml) was heated in a 16 ml Teflon-lined reaction vessel at 453 K for 5 days; the mixture was cooled to room temperature over a period of 40 h. The product was collected by filtration, washed with H_2O and air-dried.

Refinement

H atoms bonded to C atoms were placed geometrically and refined as riding atoms. The pyridyl (N1) was found from a difference Fourier map and refined as riding, with $N-H = 0.86 \text{ \AA}$, and the water H atoms were found from Fourier difference maps and refined with restraints for $O-H$ distances (0.85 \AA) with $U_{iso}(H) = 1.2U_{eq}(O)$. The highest residual electron density was found at 0.07 \AA from Nd1 atom and the deepest hole at 0.56 \AA from the O1W atom.

Figures

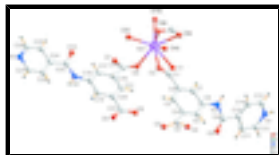


Fig. 1. The *ORTEP* drawing of the title compound (I). Displacement ellipsoids are drawn at 30% probability level. [Symmetry codes: (i) $2 - x, -1/2 + y, 3/2 - z$ (ii) $x, 1/2 - y, -1/2 + z$ (iii) $2 - x, -y, 1 - z$.]

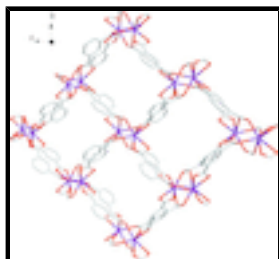


Fig. 2. Projection showing the two-dimensional structure of the compound linked by L^{2-} ; all the pyridyl groups are omitted.

Poly[[[μ_3 -5-(pyridine-4-carboxamido)isophthalato] $\{\mu_3$ -5-[(pyridin-1-ium-4-yl)carbonylamino]isophthalato}neodymium(III)] dihydrate]

Crystal data

[Nd(C₁₄H₉N₂O₅)(C₁₄H₈N₂O₅)]·2H₂O

$M_r = 749.73$

Monoclinic, $P2_1/c$

Hall symbol: -p 2ybc

$a = 13.4421$ (15) Å

$b = 13.7754$ (17) Å

$c = 16.2418$ (13) Å

$\beta = 115.813$ (4)°

$V = 2707.4$ (5) Å³

$Z = 4$

$F(000) = 1492$

$D_x = 1.839$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 8058 reflections

$\theta = 2.2$ – 28.3 °

$\mu = 2.00$ mm⁻¹

$T = 291$ K

Block, colorless

$0.18 \times 0.16 \times 0.12$ mm

Data collection

Bruker SMART APEX
diffractometer

Radiation source: fine-focus sealed tube
graphite

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 2008)

$T_{\min} = 0.715$, $T_{\max} = 0.796$

14297 measured reflections

5287 independent reflections

4792 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.052$

$\theta_{\max} = 26.0$ °, $\theta_{\min} = 1.7$ °

$h = -16 \rightarrow 15$

$k = -16 \rightarrow 16$

$l = -20 \rightarrow 16$

Refinement

Refinement on F^2

Primary atom site location: structure-invariant direct
methods

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.038$$

$$wR(F^2) = 0.101$$

$$S = 1.07$$

5287 reflections

406 parameters

0 restraints

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.055P)^2 + 3.5595P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\max} = 1.83 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -1.61 \text{ e } \text{\AA}^{-3}$$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O2W	0.4403 (3)	0.6060 (3)	0.0967 (3)	0.0456 (9)
O1W	0.4253 (3)	0.1460 (3)	0.5360 (2)	0.0537 (11)
Nd1	0.898403 (16)	0.014994 (16)	0.571291 (14)	0.01719 (10)
O1	0.7704 (2)	0.1428 (2)	0.56596 (19)	0.0250 (6)
O2	0.9065 (2)	0.1322 (2)	0.70371 (19)	0.0236 (6)
O3	0.9144 (2)	0.4679 (2)	0.8775 (2)	0.0289 (7)
O4	0.7730 (2)	0.4510 (2)	0.91087 (18)	0.0244 (6)
O5	0.4645 (3)	0.5086 (2)	0.5783 (2)	0.0356 (8)
O6	0.9932 (2)	0.1532 (2)	0.55514 (19)	0.0212 (6)
O7	1.1226 (2)	0.1364 (2)	0.50548 (19)	0.0218 (6)
O8	1.0730 (2)	0.4069 (2)	0.7959 (2)	0.0279 (7)
O9	1.2447 (2)	0.4326 (2)	0.89481 (19)	0.0255 (7)
O10	1.4746 (3)	0.0803 (2)	0.7157 (2)	0.0374 (8)
N2	0.4776 (3)	0.3440 (3)	0.5862 (2)	0.0256 (8)
H2	0.4395	0.2915	0.5689	0.031*
N1	0.0898 (3)	0.4082 (3)	0.3610 (3)	0.0343 (9)
H1	0.0259	0.3971	0.3130	0.041*
N4	1.4748 (3)	0.2155 (3)	0.7943 (2)	0.0275 (8)
H4A	1.5175	0.2570	0.8333	0.033*
N3	1.8768 (3)	0.1047 (4)	0.9233 (4)	0.0524 (13)
C1	0.8171 (3)	0.1699 (3)	0.6476 (3)	0.0189 (8)
C2	0.7617 (3)	0.2495 (3)	0.6762 (3)	0.0174 (8)
C3	0.8178 (3)	0.3080 (3)	0.7518 (3)	0.0177 (8)

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H3	0.8933	0.3002	0.7871	0.021*
C4	0.7597 (3)	0.3787 (3)	0.7744 (2)	0.0163 (8)
C5	0.6465 (3)	0.3920 (3)	0.7214 (3)	0.0202 (8)
H5	0.6080	0.4383	0.7377	0.024*
C6	0.5922 (3)	0.3350 (3)	0.6440 (3)	0.0197 (8)
C7	0.6500 (3)	0.2648 (3)	0.6220 (3)	0.0218 (8)
H7	0.6133	0.2270	0.5700	0.026*
C8	0.8192 (3)	0.4374 (3)	0.8595 (3)	0.0165 (8)
C9	0.4240 (3)	0.4284 (3)	0.5564 (3)	0.0240 (9)
C10	0.3035 (3)	0.4177 (3)	0.4879 (3)	0.0229 (9)
C11	0.2528 (4)	0.4968 (4)	0.4336 (4)	0.0423 (13)
H11	0.2917	0.5545	0.4407	0.051*
C12	0.1446 (5)	0.4904 (4)	0.3688 (4)	0.0490 (15)
H12	0.1105	0.5430	0.3312	0.059*
C13	0.1334 (4)	0.3328 (4)	0.4145 (4)	0.0414 (12)
H13	0.0912	0.2773	0.4082	0.050*
C14	0.2411 (4)	0.3362 (4)	0.4796 (3)	0.0383 (12)
H14	0.2717	0.2834	0.5180	0.046*
C15	1.0895 (3)	0.1640 (3)	0.5638 (3)	0.0178 (8)
C16	1.1716 (3)	0.2123 (3)	0.6484 (3)	0.0195 (8)
C17	1.2835 (3)	0.1920 (3)	0.6786 (3)	0.0222 (9)
H17	1.3067	0.1502	0.6455	0.027*
C18	1.3594 (3)	0.2342 (3)	0.7580 (3)	0.0254 (9)
C19	1.3240 (3)	0.2999 (3)	0.8052 (3)	0.0248 (9)
H19	1.3756	0.3302	0.8576	0.030*
C20	1.2126 (3)	0.3201 (3)	0.7746 (3)	0.0197 (8)
C21	1.1354 (3)	0.2748 (3)	0.6964 (3)	0.0212 (8)
H21	1.0603	0.2863	0.6766	0.025*
C22	1.1748 (3)	0.3909 (3)	0.8245 (3)	0.0195 (8)
C23	1.5245 (3)	0.1407 (3)	0.7749 (3)	0.0260 (9)
C24	1.6479 (3)	0.1320 (3)	0.8288 (3)	0.0244 (9)
C25	1.7001 (4)	0.0579 (4)	0.8048 (4)	0.0432 (13)
H25	1.6603	0.0165	0.7563	0.052*
C26	1.8130 (4)	0.0472 (5)	0.8552 (4)	0.0518 (15)
H26	1.8468	-0.0042	0.8402	0.062*
C27	1.8250 (4)	0.1743 (4)	0.9450 (4)	0.0453 (13)
H27	1.8669	0.2148	0.9935	0.054*
C28	1.7122 (4)	0.1909 (4)	0.9002 (3)	0.0362 (11)
H28	1.6804	0.2415	0.9183	0.043*
H1WA	0.3687	0.1203	0.4936	0.054*
H1WB	0.4382	0.1202	0.5872	0.054*
H2WA	0.4156	0.5777	0.1305	0.054*
H2WB	0.3799	0.6182	0.0501	0.054*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2W	0.0282 (18)	0.050 (2)	0.050 (2)	0.0082 (17)	0.0092 (16)	0.0003 (19)

O1W	0.056 (2)	0.050 (2)	0.033 (2)	0.025 (2)	-0.0012 (18)	0.0001 (18)
Nd1	0.01348 (13)	0.02044 (15)	0.01550 (14)	0.00045 (7)	0.00432 (10)	0.00084 (8)
O1	0.0206 (14)	0.0288 (16)	0.0190 (14)	0.0067 (12)	0.0026 (12)	-0.0077 (12)
O2	0.0143 (14)	0.0323 (16)	0.0176 (14)	0.0070 (12)	0.0008 (12)	-0.0019 (12)
O3	0.0126 (14)	0.0271 (16)	0.0389 (18)	-0.0022 (12)	0.0037 (13)	-0.0093 (14)
O4	0.0250 (15)	0.0305 (16)	0.0154 (14)	-0.0016 (13)	0.0067 (12)	-0.0050 (12)
O5	0.0210 (17)	0.0343 (19)	0.036 (2)	-0.0009 (13)	-0.0023 (15)	-0.0072 (15)
O6	0.0143 (13)	0.0190 (14)	0.0285 (15)	-0.0013 (11)	0.0076 (12)	-0.0004 (12)
O7	0.0225 (14)	0.0219 (15)	0.0213 (14)	-0.0033 (12)	0.0098 (12)	-0.0039 (12)
O8	0.0141 (14)	0.0380 (18)	0.0269 (16)	0.0020 (13)	0.0046 (12)	-0.0134 (14)
O9	0.0177 (14)	0.0329 (17)	0.0211 (15)	0.0025 (12)	0.0040 (12)	-0.0097 (13)
O10	0.0294 (17)	0.0375 (19)	0.0365 (19)	0.0018 (15)	0.0063 (15)	-0.0118 (16)
N2	0.0120 (16)	0.0273 (19)	0.0258 (19)	-0.0004 (14)	-0.0027 (14)	-0.0088 (15)
N1	0.0175 (18)	0.048 (2)	0.025 (2)	-0.0038 (17)	-0.0017 (16)	0.0043 (18)
N4	0.0122 (16)	0.037 (2)	0.030 (2)	0.0002 (15)	0.0056 (15)	-0.0132 (17)
N3	0.026 (2)	0.051 (3)	0.073 (3)	0.005 (2)	0.015 (2)	-0.008 (3)
C1	0.0180 (19)	0.0190 (19)	0.021 (2)	-0.0013 (16)	0.0092 (17)	-0.0008 (16)
C2	0.0137 (18)	0.0183 (19)	0.0177 (19)	0.0018 (15)	0.0046 (16)	-0.0015 (15)
C3	0.0101 (17)	0.023 (2)	0.0165 (19)	0.0022 (15)	0.0027 (15)	0.0013 (16)
C4	0.0112 (17)	0.0204 (19)	0.0127 (18)	-0.0007 (15)	0.0008 (15)	-0.0021 (15)
C5	0.0152 (19)	0.022 (2)	0.020 (2)	0.0030 (15)	0.0046 (16)	-0.0050 (16)
C6	0.0102 (17)	0.025 (2)	0.0173 (19)	-0.0010 (15)	-0.0002 (15)	-0.0052 (16)
C7	0.0166 (19)	0.025 (2)	0.018 (2)	-0.0007 (16)	0.0023 (16)	-0.0054 (17)
C8	0.0125 (18)	0.0145 (19)	0.0145 (18)	0.0009 (14)	-0.0016 (15)	0.0003 (15)
C9	0.0144 (19)	0.037 (3)	0.017 (2)	0.0015 (18)	0.0030 (16)	-0.0061 (18)
C10	0.0167 (19)	0.031 (2)	0.0173 (19)	0.0016 (17)	0.0040 (16)	-0.0023 (17)
C11	0.026 (3)	0.042 (3)	0.044 (3)	-0.005 (2)	0.002 (2)	0.012 (2)
C12	0.029 (3)	0.056 (4)	0.044 (3)	-0.002 (2)	-0.001 (3)	0.022 (3)
C13	0.023 (2)	0.038 (3)	0.050 (3)	-0.004 (2)	0.003 (2)	0.007 (2)
C14	0.021 (2)	0.040 (3)	0.042 (3)	0.003 (2)	0.002 (2)	0.011 (2)
C15	0.0171 (19)	0.0111 (18)	0.022 (2)	-0.0002 (14)	0.0056 (16)	0.0025 (15)
C16	0.0175 (19)	0.024 (2)	0.0174 (19)	-0.0035 (16)	0.0081 (16)	-0.0018 (16)
C17	0.0186 (19)	0.025 (2)	0.023 (2)	-0.0008 (16)	0.0091 (17)	-0.0073 (17)
C18	0.016 (2)	0.033 (2)	0.025 (2)	0.0006 (17)	0.0076 (17)	-0.0049 (19)
C19	0.018 (2)	0.032 (2)	0.022 (2)	-0.0007 (17)	0.0065 (17)	-0.0077 (18)
C20	0.0191 (19)	0.023 (2)	0.0185 (19)	0.0010 (16)	0.0098 (16)	-0.0014 (16)
C21	0.0139 (18)	0.025 (2)	0.025 (2)	-0.0012 (16)	0.0094 (17)	-0.0026 (17)
C22	0.019 (2)	0.020 (2)	0.021 (2)	-0.0006 (16)	0.0104 (17)	-0.0002 (16)
C23	0.023 (2)	0.029 (2)	0.025 (2)	-0.0020 (18)	0.0097 (19)	-0.0018 (18)
C24	0.017 (2)	0.027 (2)	0.030 (2)	0.0005 (17)	0.0117 (18)	0.0009 (18)
C25	0.032 (3)	0.047 (3)	0.049 (3)	0.002 (2)	0.016 (2)	-0.014 (3)
C26	0.029 (3)	0.055 (3)	0.071 (4)	0.010 (3)	0.021 (3)	-0.018 (3)
C27	0.026 (2)	0.041 (3)	0.055 (3)	-0.003 (2)	0.005 (2)	-0.013 (3)
C28	0.024 (2)	0.033 (3)	0.048 (3)	0.004 (2)	0.013 (2)	-0.009 (2)

Geometric parameters (Å, °)

O2W—H2WA	0.8499	C2—C3	1.385 (5)
O2W—H2WB	0.8515	C2—C7	1.386 (5)

supplementary materials

O1W—H1WA	0.8498	C3—C4	1.393 (5)
O1W—H1WB	0.8499	C3—H3	0.9300
Nd1—O6	2.368 (3)	C4—C5	1.395 (5)
Nd1—O3 ⁱ	2.371 (3)	C4—C8	1.498 (5)
Nd1—O7 ⁱⁱ	2.383 (3)	C5—C6	1.390 (5)
Nd1—O1	2.436 (3)	C5—H5	0.9300
Nd1—O4 ⁱⁱⁱ	2.455 (3)	C6—C7	1.381 (5)
Nd1—O9 ⁱ	2.493 (3)	C7—H7	0.9300
Nd1—O8 ⁱ	2.508 (3)	C9—C10	1.521 (5)
Nd1—O2	2.653 (3)	C10—C14	1.373 (6)
O1—C1	1.252 (5)	C10—C11	1.381 (7)
O2—C1	1.261 (5)	C11—C12	1.377 (8)
O3—C8	1.254 (5)	C11—H11	0.9300
O3—Nd1 ^{iv}	2.371 (3)	C12—H12	0.9300
O4—C8	1.252 (5)	C13—C14	1.372 (6)
O4—Nd1 ^v	2.455 (3)	C13—H13	0.9300
O5—C9	1.214 (5)	C14—H14	0.9300
O6—C15	1.249 (4)	C15—C16	1.493 (5)
O7—C15	1.267 (5)	C16—C21	1.384 (5)
O7—Nd1 ⁱⁱ	2.383 (3)	C16—C17	1.392 (5)
O8—C22	1.259 (5)	C17—C18	1.376 (6)
O8—Nd1 ^{iv}	2.508 (3)	C17—H17	0.9300
O9—C22	1.258 (5)	C18—C19	1.397 (6)
O9—Nd1 ^{iv}	2.493 (3)	C19—C20	1.386 (5)
O10—C23	1.226 (5)	C19—H19	0.9300
N2—C9	1.343 (6)	C20—C21	1.389 (6)
N2—C6	1.418 (5)	C20—C22	1.492 (5)
N2—H2	0.8596	C21—H21	0.9300
N1—C13	1.317 (6)	C23—C24	1.505 (6)
N1—C12	1.327 (7)	C24—C28	1.370 (6)
N1—H1	0.8864	C24—C25	1.387 (6)
N4—C23	1.339 (5)	C25—C26	1.383 (7)
N4—C18	1.422 (5)	C25—H25	0.9300
N4—H4A	0.8607	C26—H26	0.9300
N3—C27	1.319 (7)	C27—C28	1.386 (6)
N3—C26	1.326 (7)	C27—H27	0.9300
C1—C2	1.509 (5)	C28—H28	0.9300
H2WA—O2W—H2WB	100.1	C7—C6—C5	119.7 (3)
H1WA—O1W—H1WB	110.4	C7—C6—N2	117.5 (3)
O6—Nd1—O3 ⁱ	73.66 (10)	C5—C6—N2	122.7 (4)
O6—Nd1—O7 ⁱⁱ	126.34 (9)	C6—C7—C2	121.2 (4)
O3 ⁱ —Nd1—O7 ⁱⁱ	79.05 (10)	C6—C7—H7	119.4
O6—Nd1—O1	79.68 (10)	C2—C7—H7	119.4
O3 ⁱ —Nd1—O1	146.63 (10)	O4—C8—O3	123.2 (4)
O7 ⁱⁱ —Nd1—O1	133.86 (9)	O4—C8—C4	118.4 (3)

O6—Nd1—O4 ⁱⁱⁱ	83.28 (10)	O3—C8—C4	118.4 (3)
O3 ⁱ —Nd1—O4 ⁱⁱⁱ	123.93 (10)	O5—C9—N2	125.5 (4)
O7 ⁱⁱ —Nd1—O4 ⁱⁱⁱ	75.02 (10)	O5—C9—C10	120.1 (4)
O1—Nd1—O4 ⁱⁱⁱ	71.12 (10)	N2—C9—C10	114.4 (4)
O6—Nd1—O9 ⁱ	153.47 (10)	C14—C10—C11	118.2 (4)
O3 ⁱ —Nd1—O9 ⁱ	126.94 (10)	C14—C10—C9	124.2 (4)
O7 ⁱⁱ —Nd1—O9 ⁱ	77.88 (10)	C11—C10—C9	117.6 (4)
O1—Nd1—O9 ⁱ	74.69 (10)	C12—C11—C10	120.1 (5)
O4 ⁱⁱⁱ —Nd1—O9 ⁱ	94.70 (9)	C12—C11—H11	120.0
O6—Nd1—O8 ⁱ	133.14 (10)	C10—C11—H11	120.0
O3 ⁱ —Nd1—O8 ⁱ	78.10 (10)	N1—C12—C11	119.0 (5)
O7 ⁱⁱ —Nd1—O8 ⁱ	82.49 (10)	N1—C12—H12	120.5
O1—Nd1—O8 ⁱ	107.52 (10)	C11—C12—H12	120.5
O4 ⁱⁱⁱ —Nd1—O8 ⁱ	143.44 (10)	N1—C13—C14	119.9 (5)
O9 ⁱ —Nd1—O8 ⁱ	51.99 (9)	N1—C13—H13	120.1
O6—Nd1—O2	76.49 (9)	C14—C13—H13	120.1
O3 ⁱ —Nd1—O2	102.66 (10)	C13—C14—C10	119.9 (5)
O7 ⁱⁱ —Nd1—O2	155.57 (9)	C13—C14—H14	120.1
O1—Nd1—O2	50.83 (9)	C10—C14—H14	120.1
O4 ⁱⁱⁱ —Nd1—O2	120.74 (9)	O6—C15—O7	124.3 (4)
O9 ⁱ —Nd1—O2	82.03 (10)	O6—C15—C16	118.1 (3)
O8 ⁱ —Nd1—O2	74.19 (11)	O7—C15—C16	117.6 (3)
O6—Nd1—C22 ⁱ	152.25 (11)	C21—C16—C17	121.2 (4)
O3 ⁱ —Nd1—C22 ⁱ	102.40 (11)	C21—C16—C15	119.7 (3)
O7 ⁱⁱ —Nd1—C22 ⁱ	78.11 (10)	C17—C16—C15	119.1 (3)
O1—Nd1—C22 ⁱ	91.90 (11)	C18—C17—C16	119.5 (4)
O4 ⁱⁱⁱ —Nd1—C22 ⁱ	119.15 (10)	C18—C17—H17	120.3
O9 ⁱ —Nd1—C22 ⁱ	25.98 (10)	C16—C17—H17	120.3
O8 ⁱ —Nd1—C22 ⁱ	26.04 (10)	C17—C18—C19	119.8 (4)
O2—Nd1—C22 ⁱ	77.73 (10)	C17—C18—N4	122.8 (4)
C1—O1—Nd1	99.1 (2)	C19—C18—N4	117.4 (4)
C1—O2—Nd1	88.6 (2)	C20—C19—C18	120.4 (4)
C8—O3—Nd1 ^{iv}	172.9 (3)	C20—C19—H19	119.8
C8—O4—Nd1 ^v	115.1 (2)	C18—C19—H19	119.8
C15—O6—Nd1	131.7 (2)	C19—C20—C21	119.9 (4)
C15—O7—Nd1 ⁱⁱ	130.8 (2)	C19—C20—C22	120.4 (4)
C22—O8—Nd1 ^{iv}	93.0 (2)	C21—C20—C22	119.7 (3)
C22—O9—Nd1 ^{iv}	93.8 (2)	C16—C21—C20	119.2 (4)
C9—N2—C6	124.9 (4)	C16—C21—H21	120.4
C9—N2—H2	117.5	C20—C21—H21	120.4
C6—N2—H2	117.6	O9—C22—O8	121.1 (4)
C13—N1—C12	122.8 (4)	O9—C22—C20	119.7 (3)

supplementary materials

C13—N1—H1	115.2	O8—C22—C20	119.1 (4)
C12—N1—H1	121.1	O9—C22—Nd1 ^{iv}	60.3 (2)
C23—N4—C18	127.4 (4)	O8—C22—Nd1 ^{iv}	61.0 (2)
C23—N4—H4A	116.3	O10—C23—N4	123.2 (4)
C18—N4—H4A	116.3	O10—C23—C24	119.3 (4)
C27—N3—C26	115.5 (4)	N4—C23—C24	117.4 (4)
O1—C1—O2	121.5 (4)	C28—C24—C25	117.6 (4)
O1—C1—C2	117.1 (3)	C28—C24—C23	125.0 (4)
O2—C1—C2	121.3 (3)	C25—C24—C23	117.3 (4)
C3—C2—C7	119.7 (4)	C26—C25—C24	118.1 (5)
C3—C2—C1	123.0 (3)	C26—C25—H25	121.0
C7—C2—C1	117.3 (3)	C24—C25—H25	121.0
C2—C3—C4	119.2 (3)	N3—C26—C25	125.1 (5)
C2—C3—H3	120.4	N3—C26—H26	117.5
C4—C3—H3	120.4	C25—C26—H26	117.5
C3—C4—C5	121.0 (3)	N3—C27—C28	124.4 (5)
C3—C4—C8	119.0 (3)	N3—C27—H27	117.8
C5—C4—C8	119.9 (3)	C28—C27—H27	117.8
C6—C5—C4	119.1 (4)	C24—C28—C27	119.3 (4)
C6—C5—H5	120.5	C24—C28—H28	120.4
C4—C5—H5	120.5	C27—C28—H28	120.4
O6—Nd1—O1—C1	-81.7 (2)	N2—C9—C10—C14	22.5 (6)
O3 ⁱ —Nd1—O1—C1	-44.5 (3)	O5—C9—C10—C11	18.0 (6)
O7 ⁱⁱ —Nd1—O1—C1	146.9 (2)	N2—C9—C10—C11	-160.5 (5)
O4 ⁱⁱⁱ —Nd1—O1—C1	-168.1 (3)	C14—C10—C11—C12	-4.3 (9)
O9 ⁱ —Nd1—O1—C1	91.3 (3)	C9—C10—C11—C12	178.4 (5)
O8 ⁱ —Nd1—O1—C1	50.4 (3)	C13—N1—C12—C11	2.1 (9)
O2—Nd1—O1—C1	-0.8 (2)	C10—C11—C12—N1	1.3 (10)
C22 ⁱ —Nd1—O1—C1	71.7 (3)	C12—N1—C13—C14	-2.4 (9)
O6—Nd1—O2—C1	88.4 (2)	N1—C13—C14—C10	-0.8 (8)
O3 ⁱ —Nd1—O2—C1	157.9 (2)	C11—C10—C14—C13	4.1 (8)
O7 ⁱⁱ —Nd1—O2—C1	-110.8 (3)	C9—C10—C14—C13	-178.9 (5)
O1—Nd1—O2—C1	0.8 (2)	Nd1—O6—C15—O7	75.6 (5)
O4 ⁱⁱⁱ —Nd1—O2—C1	14.8 (3)	Nd1—O6—C15—C16	-104.1 (4)
O9 ⁱ —Nd1—O2—C1	-75.9 (2)	Nd1 ⁱⁱ —O7—C15—O6	-48.7 (5)
O8 ⁱ —Nd1—O2—C1	-128.6 (2)	Nd1 ⁱⁱ —O7—C15—C16	131.1 (3)
C22 ⁱ —Nd1—O2—C1	-102.0 (2)	O6—C15—C16—C21	-24.5 (5)
O3 ⁱ —Nd1—O6—C15	8.1 (3)	O7—C15—C16—C21	155.8 (4)
O7 ⁱⁱ —Nd1—O6—C15	-54.4 (4)	O6—C15—C16—C17	154.2 (4)
O1—Nd1—O6—C15	167.8 (4)	O7—C15—C16—C17	-25.5 (5)
O4 ⁱⁱⁱ —Nd1—O6—C15	-120.2 (3)	C21—C16—C17—C18	0.4 (6)
O9 ⁱ —Nd1—O6—C15	152.7 (3)	C15—C16—C17—C18	-178.3 (4)
O8 ⁱ —Nd1—O6—C15	63.4 (4)	C16—C17—C18—C19	-2.6 (7)
O2—Nd1—O6—C15	115.9 (3)	C16—C17—C18—N4	178.6 (4)
C22 ⁱ —Nd1—O6—C15	93.7 (4)	C23—N4—C18—C17	-18.5 (7)

Nd1—O1—C1—O2	1.5 (4)	C23—N4—C18—C19	162.6 (4)
Nd1—O1—C1—C2	-178.3 (3)	C17—C18—C19—C20	2.4 (7)
Nd1—O2—C1—O1	-1.4 (4)	N4—C18—C19—C20	-178.8 (4)
Nd1—O2—C1—C2	178.5 (3)	C18—C19—C20—C21	0.1 (7)
O1—C1—C2—C3	-158.1 (4)	C18—C19—C20—C22	-179.4 (4)
O2—C1—C2—C3	22.1 (6)	C17—C16—C21—C20	2.0 (6)
O1—C1—C2—C7	20.5 (5)	C15—C16—C21—C20	-179.3 (4)
O2—C1—C2—C7	-159.4 (4)	C19—C20—C21—C16	-2.2 (6)
C7—C2—C3—C4	2.7 (6)	C22—C20—C21—C16	177.3 (4)
C1—C2—C3—C4	-178.8 (4)	Nd1 ^{iv} —O9—C22—O8	4.0 (4)
C2—C3—C4—C5	-0.8 (6)	Nd1 ^{iv} —O9—C22—C20	-175.0 (3)
C2—C3—C4—C8	176.1 (4)	Nd1 ^{iv} —O8—C22—O9	-4.0 (4)
C3—C4—C5—C6	-1.5 (6)	Nd1 ^{iv} —O8—C22—C20	175.1 (3)
C8—C4—C5—C6	-178.3 (4)	C19—C20—C22—O9	0.1 (6)
C4—C5—C6—C7	1.7 (6)	C21—C20—C22—O9	-179.4 (4)
C4—C5—C6—N2	-179.8 (4)	C19—C20—C22—O8	-179.0 (4)
C9—N2—C6—C7	-136.0 (4)	C21—C20—C22—O8	1.5 (6)
C9—N2—C6—C5	45.4 (6)	C18—N4—C23—O10	5.7 (7)
C5—C6—C7—C2	0.3 (6)	C18—N4—C23—C24	-174.7 (4)
N2—C6—C7—C2	-178.3 (4)	O10—C23—C24—C28	-175.8 (5)
C3—C2—C7—C6	-2.5 (6)	N4—C23—C24—C28	4.6 (7)
C1—C2—C7—C6	178.9 (4)	O10—C23—C24—C25	3.3 (6)
Nd1 ^v —O4—C8—O3	-22.1 (5)	N4—C23—C24—C25	-176.3 (4)
Nd1 ^v —O4—C8—C4	155.6 (3)	C28—C24—C25—C26	0.9 (8)
C3—C4—C8—O4	-135.3 (4)	C23—C24—C25—C26	-178.3 (5)
C5—C4—C8—O4	41.6 (5)	C27—N3—C26—C25	2.8 (10)
C3—C4—C8—O3	42.5 (5)	C24—C25—C26—N3	-2.4 (10)
C5—C4—C8—O3	-140.6 (4)	C26—N3—C27—C28	-1.8 (9)
C6—N2—C9—O5	-4.0 (7)	C25—C24—C28—C27	-0.1 (7)
C6—N2—C9—C10	174.4 (4)	C23—C24—C28—C27	179.1 (5)
O5—C9—C10—C14	-159.0 (5)	N3—C27—C28—C24	0.5 (9)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2 \cdots O1W	0.86	2.06	2.846 (5)	151.
N1—H1 \cdots O2 ^{vi}	0.89	1.85	2.725 (4)	171.
O1W—H1WA \cdots O9 ^{vi}	0.85	1.89	2.734 (5)	177.
O2W—H2WA \cdots O10 ^{vi}	0.85	2.52	3.124 (5)	129.
O1W—H1WB \cdots O10 ^{vii}	0.85	2.00	2.845 (5)	171.
O2W—H2WA \cdots O4 ^{viii}	0.85	2.36	2.923 (5)	125.
O2W—H2WB \cdots O1 ^{ix}	0.85	2.11	2.957 (4)	178.
N4—H4A \cdots O2W ^x	0.86	2.15	2.953 (5)	156.

Symmetry codes: (vi) $x-1, -y+1/2, z-1/2$; (vii) $x-1, y, z$; (viii) $-x+1, -y+1, -z+1$; (ix) $-x+1, y+1/2, -z+1/2$; (x) $-x+2, -y+1, -z+1$.

Fig. 1

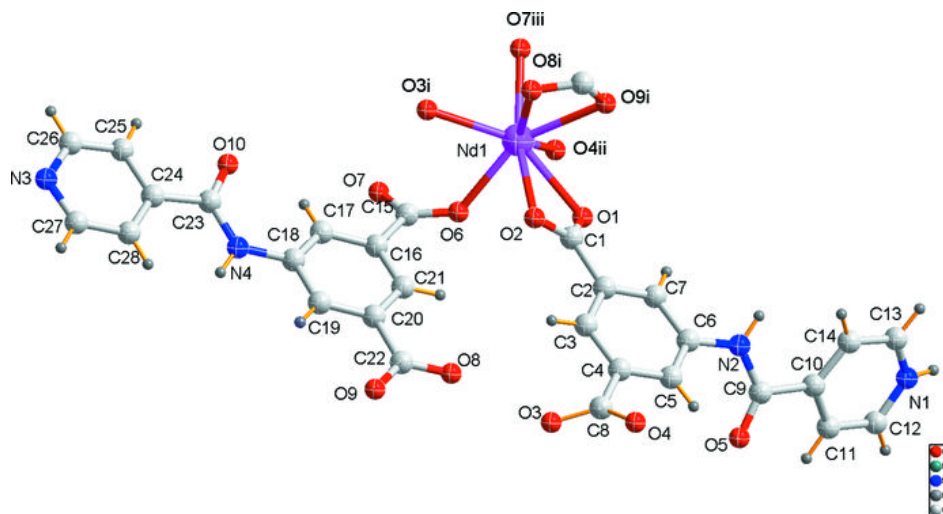


Fig. 2

