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# Tris(piperazinediium) bis[tris(pyridine-2,6-dicarboxylato)neodymate(III)] 15.33-hydrate

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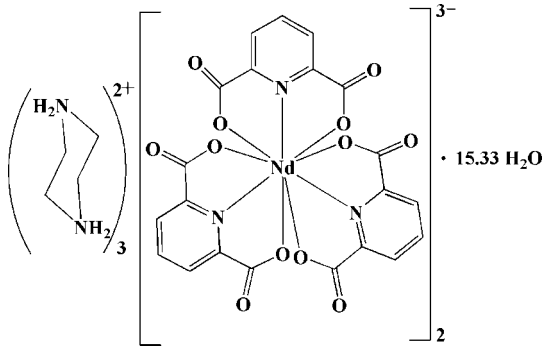
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.009 Å; disorder in solvent or counterion; $R$ factor = 0.064; $wR$ factor = 0.199; data-to-parameter ratio = 17.9.

The title compound, $(C_4H_{12}N_2)_3[Nd(C_7H_3NO_4)_3]_2\cdot 15.33H_2O$ or $(pipzH_2)_3[Nd(pydc)_3]_2\cdot 15.33H_2O$ (in which pipz is piperazine and $pydcH_2$ is pyridine-2,6-dicarboxylic acid), was synthesized by the reaction of $NdCl_3\cdot 6H_2O$ with the proton-transfer compound $(pipzH_2)(pydc)$ in aqueous solution. The nine donor atoms of the three $pydc^{2-}$ ligands form a distorted tricapped trigonal–prismatic arrangement around the $Nd^{III}$ center. Considerable C—O$\cdots\pi$ stacking interactions between CO groups of carboxylate fragments and aromatic rings of $pydc^{2-}$ with distances of 3.135 (5)–3.255 (5) Å are observed. In the crystal structure, a wide range of hydrogen-bonding [of the types O—H$\cdots$O, N—H$\cdots$O and C—H$\cdots$O, with $D\cdots A$ distances ranging from 2.608 (10) to 3.278 (7) Å], ion-pairing and C—O$\cdots\pi$ stacking interactions connect the various components into a supramolecular structure. There is a high degree of solvent disorder in the structure; the occupancies of five water molecules refined to 0.6, 0.5, 0.4, 0.25 and 0.25.

## Related literature

For related literature, see: Aghabozorg, Attar Gharamaleki, Ghadermazi *et al.* (2007); Aghabozorg, Attar Gharamaleki, Ghasemikhah *et al.* (2007); Aghabozorg, Daneshvar *et al.* (2007). For synthesis, see: Aghabozorg *et al.* (2006).




## Experimental

### Crystal data

$(C_4H_{12}N_2)_3[Nd(C_7H_3NO_4)_3]_2\cdot 15.33H_2O$
$M_r$ = 915.24
Trigonal, $P\overline{3}$
$a$ = 25.5045 (9) Å
$c$ = 10.0984 (7) Å
$V$ = 5688.7 (5) Å$^3$
$Z$ = 6
Mo $K\alpha$ radiation
$\mu$ = 1.46 mm$^{-1}$
$T$ = 100 (2) K
0.49 × 0.18 × 0.08 mm

### Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*APEX2*; Bruker, 2005)
$T_{min}$ = 0.536, $T_{max}$ = 0.892
64426 measured reflections
9135 independent reflections
7444 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.055

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.198
$S$ = 1.02
9135 reflections
509 parameters
18 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 4.81 e Å$^{-3}$
$\Delta\rho_{min}$ = −2.19 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Nd1—O3 | 2.465 (4) | Nd1—O11 | 2.487 (4) |
| Nd1—O7 | 2.467 (4) | Nd1—N2 | 2.565 (4) |
| Nd1—O5 | 2.483 (4) | Nd1—N1 | 2.568 (4) |
| Nd1—O1 | 2.482 (4) | Nd1—N3 | 2.575 (4) |
| Nd1—O9 | 2.485 (4) | | |
| N2—Nd1—N1 | 118.22 (12) | N1—Nd1—N3 | 120.15 (12) |
| N2—Nd1—N3 | 121.23 (12) | | |

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

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| N2*S*—H3$\cdots$O4 | 0.92 | 1.97 | 2.804 (6) | 151 |
| N2*S*—H4$\cdots$O6$^{i}$ | 0.92 | 1.92 | 2.760 (6) | 151 |
| N3*S*—H5$\cdots$O10$^{ii}$ | 0.92 | 1.95 | 2.780 (6) | 150 |
| N3*S*—H6$\cdots$O8$^{iii}$ | 0.92 | 1.99 | 2.836 (6) | 153 |
| N1*S*—H1$\cdots$O2$^{iv}$ | 0.92 | 1.92 | 2.758 (6) | 150 |
| N1*S*—H2$\cdots$O12$^{v}$ | 0.92 | 2.03 | 2.874 (6) | 153 |
| O1*W*—H7$\cdots$O9$^{ii}$ | 0.85 | 2.03 | 2.863 (7) | 167 |
| O1*W*—H8$\cdots$O9*W*$^{vi}$ | 0.85 | 2.00 | 2.838 (17) | 166 |
| O2*W*—H9$\cdots$O1$^{iv}$ | 0.85 | 2.43 | 3.278 (7) | 180 |
| O2*W*—H10$\cdots$O1*W*$^{ii}$ | 0.85 | 2.17 | 3.024 (8) | 180 |
| O3*W*—H11$\cdots$O5$^{i}$ | 0.85 | 2.14 | 2.970 (7) | 167 |
| O3*W*—H12$\cdots$O6*W*$^{i}$ | 0.85 | 2.11 | 2.950 (11) | 171 |
| O4*W*—H13$\cdots$O1$^{iv}$ | 0.85 | 2.04 | 2.878 (7) | 166 |

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| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O4$W$—H14···O8$W^{iv}$ | 0.85 | 2.17 | 2.985 (14) | 162 |
| O5$W$—H15···O4$^{vii}$ | 0.85 | 1.82 | 2.674 (6) | 180 |
| O5$W$—H16···O6$W^{viii}$ | 0.85 | 1.95 | 2.804 (9) | 179 |
| O6$W$—H17···O10$W$ | 0.85 | 2.13 | 2.972 (10) | 174 |
| O6$W$—H18···O12$^{vii}$ | 0.85 | 2.16 | 3.011 (10) | 180 |
| O7$W$—H19···O8$^{vii}$ | 0.85 | 1.76 | 2.608 (10) | 179 |
| O7$W$—H20···O7$W^{ix}$ | 0.85 | 1.95 | 2.797 (11) | 177 |
| O8$W$—H21···O5$W$ | 0.85 | 1.99 | 2.787 (12) | 156 |
| O8$W$—H22···O5$W^{x}$ | 0.85 | 1.87 | 2.723 (11) | 180 |
| O9$W$—H23···O8$^{xi}$ | 0.85 | 2.29 | 3.136 (18) | 179 |
| O9$W$—H24···O7$W$ | 0.85 | 1.92 | 2.765 (20) | 179 |
| C3$S$—H3Sc···O10$^{ii}$ | 0.99 | 2.49 | 3.200 (8) | 129 |
| C4$S$—H4$SB$···O6$^{i}$ | 0.99 | 2.47 | 3.179 (8) | 128 |
| C4$S$—H4$SB$···O7$^{iii}$ | 0.99 | 2.51 | 3.076 (9) | 116 |
| C5$S$—H5$SA$···O6$^{i}$ | 0.99 | 2.46 | 3.180 (8) | 129 |
| C6$S$—H6$SB$···O3 | 0.99 | 2.52 | 3.092 (8) | 116 |
| C6$S$—H6$SB$···O10$^{ii}$ | 0.99 | 2.45 | 3.169 (7) | 129 |
| C1$S$—H1$SC$···O2$^{xii}$ | 0.99 | 2.43 | 3.151 (7) | 129 |
| C2$S$—H2$SB$···O11$^{v}$ | 0.99 | 2.55 | 3.112 (8) | 116 |
| C2$S$—H2$SB$···O2$^{xii}$ | 0.99 | 2.49 | 3.194 (7) | 128 |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *APEX2*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2054).

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# supplementary materials

*Acta Cryst.* (2008). E**64**, m350-m351 [ **doi:10.1107/S1600536807068328** ]

# Tris(piperazinediium) bis[tris(pyridine-2,6-dicarboxylato)neodymate(III)] 15.33-hydrate

**Z. Derikvand, H. Aghabozorg, A. Nemati, M. Ghadermazi and J. Attar Gharamaleki**

## Comment

Recently, we have defined a plan to prepare water soluble proton transfer compounds as novel self assembled systems that can function as suitable ligands in the synthesis of metal complexes. In this regard, we have reported cases in which proton transfers from pyridine-2,6-dicarboxylic acid, $pydcH_2$, and benzene-1,2,4,5-tetracarboxylicacid, $btcH_4$, to propane-1,3-diamine (pn) and 1,10-phenanthroline, (phen), resulted in the formation of novel self-assembled $(pnH_2)(pydc).(pydcH_2)\cdot 2.5H_2O$, $(pnH_2)_2(btc)\cdot 2H_2O$ and $(phenH)_4(btcH_3)_2(btcH_2)$ systems, respectively. The resulting compounds with some remaining sites as electron donors can coordinate to metallic ions (Aghabozorg, Attar Gharamaleki, Ghadermazi *et al.*, 2007; Aghabozorg, Attar Gharamaleki, Ghasemikhah *et al.*, 2007; Aghabozorg, Daneshvar *et al.*, 2007, and references therein).

Here, we report on the synthesis and X-ray crystal structure of the title compound. The molecular structure of the title compound (I) is presented in Fig. 1. Selected bond lengths and angles of the structure (I) are presented in Table 1. Hydrogen bond lengths are given separately in Table 2.

In structure (I), $Nd^{III}$ is coordinated by three $(pydc)^{2-}$ groups as tridentate ligands and a nine coordinated complex results. For balancing the anionic complex, protonated piperazine, $(pipzH_2)^{2+}$, exists. The sum of bond angles, N1—Nd1—N2, N1—Nd1—N3 and N2—Nd1—N3 equals to 359.60 (12)° and indicates that Nd1 is located in the center of N1N2N3 plane. The three O atoms O1, O5 and O9 form a triangle and the other three, O3, O7 and O11 form another triangle around the $Nd^{III}$. So a distorted tricapped prism polyhedron is proposed.

Moreover, there are uncoordinated water molecules which are involved in the formation of hydrogen bonds. These water molecules form six members cyclic rings with acyclic side chains water clusters (Fig. 2).

A noticeable feature of the title compound is the presence of C—O···π stacking interactions between CO group of carboxylates with aromatic rings of $(pydc)^{2-}$ units. The C—O···π distances (measured to the center of phenyl ring) are 3.134 (5) Å for C6—O2···*Cg*1 (1 + *x*, *y*, *z*), 3.245 (5) for C21—O10···*Cg*2 (*x, y, z*) and 3.255 (5) Å for C13—O6···*Cg*3, [*Cg*1, *Cg*2 and *Cg*3 are the centroids of N1/C1—C5, N2/C8—C12 and N3/C15—C19 rings, respectively] (Fig. 3).

In the crystal structure, a wide range of non-covalent interactions consisting of hydrogen bonding (of the type O—H···O, N—H···O and C—H···O with D···A ranging from 2.608 (10) to 3.278 (7) Å, ion pairing and C—O···π stacking connect the various components into a supramolecular structure (Table 2, Fig. 4).

## Experimental

The proton transfer compound of $(pipzH_2)(pydc)$ was prepared according to our reported procedure (Aghabozorg *et al.*, 2006). A solution of $NdCl_3\cdot 6H_2O$ (320 mg, 1 mmol) in water (15 ml) was added to a solution of a solution of proton transfer

compound ($pipzH_2$)(pydc) (500 mg, 2 mmol) in water (10 ml) in a 1:2 molar ratio. Colorless crystals of (I) suitable for X-ray characterization were obtained after a few days at room temperature.

### Refinement

There is a high positive residual density of 4.81 e $Å^{-3}$ at 0.63 Å near the Nd1 center due to considerable absorption effects which could not be completely corrected.

There is a high solvent disorder in the structure. Occupancies of water molecules O7W, O8W, O9W, O10W and O11W were found out by refinement of occupation factors for this molecules as free variables and are equal to 0.6, 1/2, 0.4, 0.25 and 1/4, respectively.

The hydrogen atoms positions on carbon atoms were found geometrically. The hydrogen atoms on ordered water molecules and nitrogen atoms were found from difference Fourier maps, and for disordered water molecules were not located. All hydrogen atoms were treated in riding model with the $U_{iso}$(H) parameters equal to 1.2 $U_{eq}$(C), 1.2 $U_{eq}$(N) and 1.5 $U_{eq}$(O) where $U_{eq}$(C), $U_{eq}$(N) and $U_{eq}$(O) are the equivalent thermal parameters of the atoms to which corresponding H atoms are bonded.

### Figures

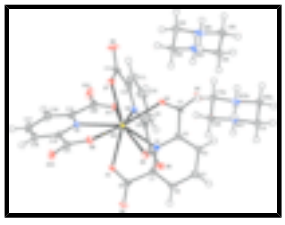

Fig. 1. The molecular structure of (I), displacement ellipsoids are drawn at the 50% probability level. Water molecules are omitted for clarity.

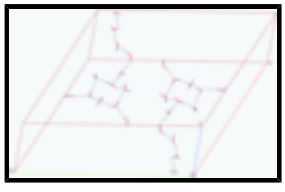

Fig. 2. C—O···π stacking interactions between CO groups of carboxylates with aromatic rings of $(pydc)^{2-}$ units. The C—O···π distances (measured to the center of phenyl ring) are 3.134 (5) Å for C6—O2···*Cg*1 (1 + *x*, *y*, *z*), 3.245 (5) Å for C21—O10···*Cg*2 (*x*, *y*, *z*) and 3.255 (5) Å for C13—O6···*Cg*3, [*Cg*1, *Cg*2 and *Cg*3 are the centroids of N1/C1—C5, N2/C8—C12 and N3/C15—C19 rings, respectively].

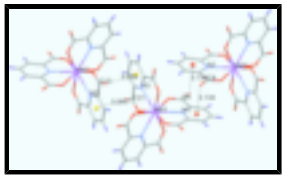

Fig. 3. Six membrane cyclic rings with acyclic side chains water clusters in the compound (I).

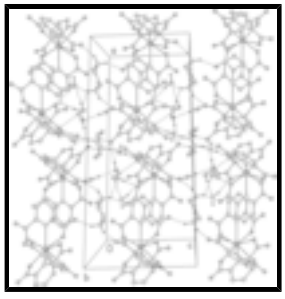

Fig. 4. The crystal packing of the compound (I), hydrogen bonds are shown as dashed lines.

**Tris(piperazinediium) bis[tris(pyridine-2,6-dicarboxylato)neodymiumate(III)] 15.33-hydrate**

*Crystal data*

| | |
|---|---|
| $(C_4H_{12}N_2)_3[Nd(C_7H_3NO_4)_3]_2{\cdot}15.33H_2O$ | $Z = 6$ |
| $M_r = 915.24$ | $F_{000} = 2798$ |
| Trigonal, $P\bar{3}$ | $D_x = 1.603$ Mg m$^{-3}$ |
| Hall symbol: -P 3 | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $a = 25.5045$ (9) Å | Cell parameters from 9656 reflections |
| $b = 25.5045$ (9) Å | $\theta = 2.6$–$31.5$º |
| $c = 10.0984$ (7) Å | $\mu = 1.46$ mm$^{-1}$ |
| $\alpha = 90$º | $T = 100$ (2) K |
| $\beta = 90$º | Prism, pink |
| $\gamma = 120$º | 0.49 × 0.18 × 0.08 mm |
| $V = 5688.7$ (5) Å$^3$ | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEXII CCD area-detector diffractometer | 9135 independent reflections |
| Radiation source: fine-focus sealed tube | 7444 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.055$ |
| $T = 100$(2) K | $\theta_{max} = 28.0$º |
| ω scans | $\theta_{min} = 1.6$º |
| Absorption correction: multi-scan (APEX2; Bruker, 2005) | $h = -33\rightarrow33$ |
| $T_{min} = 0.536$, $T_{max} = 0.892$ | $k = -33\rightarrow33$ |
| 64426 measured reflections | $l = -13\rightarrow13$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: difference Fourier map |
| $R[F^2 > 2\sigma(F^2)] = 0.063$ | H-atom parameters constrained |
| $wR(F^2) = 0.198$ | $w = 1/[\sigma^2(F_o^2) + (0.12P)^2 + 36P]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.02$ | $(\Delta/\sigma)_{max} = 0.001$ |
| 9135 reflections | $\Delta\rho_{max} = 4.81$ e Å$^{-3}$ |
| 509 parameters | $\Delta\rho_{min} = -2.19$ e Å$^{-3}$ |
| 18 restraints | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*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 $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on $F^2$ are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| Nd1 | 0.331570 (11) | −0.005899 (11) | 0.42662 (3) | 0.01072 (12) | |
| O1 | 0.38216 (18) | −0.03403 (16) | 0.5997 (4) | 0.0180 (8) | |
| O2 | 0.46277 (18) | −0.01274 (17) | 0.7241 (4) | 0.0169 (8) | |
| O3 | 0.38330 (17) | 0.07347 (16) | 0.2577 (4) | 0.0148 (7) | |
| O4 | 0.46472 (18) | 0.14173 (18) | 0.1487 (4) | 0.0186 (8) | |
| O5 | 0.36067 (18) | 0.07296 (17) | 0.5999 (4) | 0.0172 (8) | |
| O6 | 0.3421 (2) | 0.13558 (18) | 0.7208 (4) | 0.0222 (9) | |
| O7 | 0.25222 (18) | −0.03294 (16) | 0.2577 (4) | 0.0165 (8) | |
| O8 | 0.1863 (2) | −0.0172 (2) | 0.1468 (4) | 0.0303 (11) | |
| O9 | 0.25092 (18) | −0.05731 (17) | 0.5959 (4) | 0.0178 (8) | |
| O10 | 0.1930 (2) | −0.13711 (18) | 0.7233 (4) | 0.0234 (9) | |
| O11 | 0.36004 (17) | −0.05794 (17) | 0.2571 (4) | 0.0162 (7) | |
| O12 | 0.3501 (2) | −0.13946 (19) | 0.1546 (4) | 0.0297 (10) | |
| N1 | 0.4476 (2) | 0.05714 (18) | 0.4369 (4) | 0.0114 (8) | |
| N2 | 0.2714 (2) | 0.05001 (18) | 0.4347 (4) | 0.0133 (8) | |
| N3 | 0.27787 (19) | −0.12230 (19) | 0.4359 (4) | 0.0126 (8) | |
| C1 | 0.4768 (2) | 0.0497 (2) | 0.5382 (5) | 0.0132 (9) | |
| C2 | 0.5381 (2) | 0.0882 (2) | 0.5601 (5) | 0.0158 (10) | |
| H2A | 0.5577 | 0.0822 | 0.6338 | 0.019* | |
| C3 | 0.5706 (3) | 0.1360 (3) | 0.4715 (6) | 0.0195 (11) | |
| H3A | 0.6125 | 0.1633 | 0.4843 | 0.023* | |
| C4 | 0.5401 (2) | 0.1427 (2) | 0.3642 (5) | 0.0170 (10) | |
| H4A | 0.5610 | 0.1741 | 0.3013 | 0.020* | |
| C5 | 0.4785 (2) | 0.1026 (2) | 0.3516 (5) | 0.0126 (9) | |
| C6 | 0.4374 (3) | −0.0035 (2) | 0.6287 (5) | 0.0144 (10) | |
| C7 | 0.4391 (2) | 0.1062 (2) | 0.2437 (5) | 0.0126 (9) | |
| C8 | 0.2805 (3) | 0.0888 (2) | 0.5331 (5) | 0.0159 (10) | |
| C9 | 0.2451 (3) | 0.1149 (3) | 0.5509 (6) | 0.0232 (12) | |
| H9A | 0.2520 | 0.1415 | 0.6229 | 0.028* | |
| C10 | 0.1985 (3) | 0.1010 (3) | 0.4598 (6) | 0.0268 (13) | |
| H10A | 0.1731 | 0.1181 | 0.4688 | 0.032* | |
| C11 | 0.1901 (3) | 0.0619 (3) | 0.3566 (6) | 0.0242 (12) | |
| H11A | 0.1592 | 0.0523 | 0.2928 | 0.029* | |

| | | | | |
|---|---|---|---|---|
| C12 | 0.2269 (3) | 0.0370 (2) | 0.3475 (5) | 0.0164 (10) |
| C13 | 0.3317 (3) | 0.1005 (2) | 0.6274 (5) | 0.0160 (10) |
| C14 | 0.2214 (3) | −0.0082 (2) | 0.2421 (5) | 0.0185 (11) |
| C15 | 0.2920 (2) | −0.1530 (2) | 0.3489 (5) | 0.0150 (10) |
| C16 | 0.2680 (3) | −0.2154 (2) | 0.3588 (6) | 0.0207 (11) |
| H16A | 0.2778 | −0.2366 | 0.2954 | 0.025* |
| C17 | 0.2292 (3) | −0.2458 (2) | 0.4636 (6) | 0.0241 (12) |
| H17A | 0.2127 | −0.2882 | 0.4736 | 0.029* |
| C18 | 0.2145 (3) | −0.2137 (2) | 0.5542 (6) | 0.0208 (11) |
| H18A | 0.1881 | −0.2335 | 0.6265 | 0.025* |
| C19 | 0.2398 (2) | −0.1517 (2) | 0.5348 (5) | 0.0145 (10) |
| C20 | 0.3377 (2) | −0.1140 (2) | 0.2444 (5) | 0.0169 (10) |
| C21 | 0.2260 (2) | −0.1127 (2) | 0.6265 (5) | 0.0144 (10) |
| N2S | 0.3770 (2) | 0.1421 (2) | −0.0182 (5) | 0.0184 (9) |
| H3 | 0.4037 | 0.1313 | 0.0164 | 0.022* |
| H4 | 0.3743 | 0.1348 | −0.1079 | 0.022* |
| N3S | 0.2955 (2) | 0.1879 (2) | 0.0138 (5) | 0.0201 (10) |
| H5 | 0.2980 | 0.1953 | 0.1034 | 0.024* |
| H6 | 0.2688 | 0.1984 | −0.0215 | 0.024* |
| C3S | 0.4008 (3) | 0.2077 (2) | 0.0051 (6) | 0.0184 (11) |
| H3SB | 0.4404 | 0.2315 | −0.0397 | 0.022* |
| H3SC | 0.4071 | 0.2163 | 0.1012 | 0.022* |
| C4S | 0.3567 (3) | 0.2264 (3) | −0.0477 (6) | 0.0191 (11) |
| H4SA | 0.3718 | 0.2695 | −0.0268 | 0.023* |
| H4SB | 0.3536 | 0.2216 | −0.1451 | 0.023* |
| C5S | 0.2722 (3) | 0.1220 (3) | −0.0092 (6) | 0.0228 (12) |
| H5SA | 0.2662 | 0.1133 | −0.1053 | 0.027* |
| H5SB | 0.2326 | 0.0980 | 0.0353 | 0.027* |
| C6S | 0.3159 (3) | 0.1037 (2) | 0.0434 (5) | 0.0190 (11) |
| H6SA | 0.3008 | 0.0605 | 0.0229 | 0.023* |
| H6SB | 0.3190 | 0.1086 | 0.1408 | 0.023* |
| N1S | 0.0447 (2) | 0.4816 (2) | 0.9869 (4) | 0.0171 (9) |
| H1 | 0.0448 | 0.4761 | 0.8969 | 0.020* |
| H2 | 0.0726 | 0.4732 | 1.0242 | 0.020* |
| C1S | 0.0629 (2) | 0.5463 (2) | 1.0145 (5) | 0.0172 (10) |
| H1SB | 0.1030 | 0.5733 | 0.9741 | 0.021* |
| H1SC | 0.0666 | 0.5534 | 1.1113 | 0.021* |
| C2S | −0.0168 (3) | 0.4387 (2) | 1.0410 (5) | 0.0176 (11) |
| H2SB | −0.0161 | 0.4417 | 1.1388 | 0.021* |
| H2SC | −0.0283 | 0.3967 | 1.0169 | 0.021* |
| O1W | 0.2187 (3) | 0.2131 (3) | 0.1999 (5) | 0.0417 (13) |
| H7 | 0.2406 | 0.2202 | 0.2684 | 0.063* |
| H8 | 0.1816 | 0.1861 | 0.2057 | 0.063* |
| O2W | 0.0045 (2) | 0.3336 (2) | 0.8678 (6) | 0.0386 (14) |
| H9 | 0.0120 | 0.3551 | 0.7985 | 0.058* |
| H10 | 0.0048 | 0.3013 | 0.8491 | 0.058* |
| O3W | 0.4578 (3) | 0.1242 (3) | −0.1969 (5) | 0.0473 (15) |
| H11 | 0.4346 | 0.1133 | −0.2640 | 0.071* |
| H12 | 0.4951 | 0.1500 | −0.2075 | 0.071* |

| | | | | | |
|---|---|---|---|---|---|
| O4W | 0.1237 (3) | 0.4570 (4) | 0.8062 (5) | 0.0565 (18) | |
| H13 | 0.1017 | 0.4500 | 0.7380 | 0.085* | |
| H14 | 0.1567 | 0.4893 | 0.7897 | 0.085* | |
| O5W | 0.4304 (2) | −0.2332 (2) | 0.9394 (5) | 0.0404 (12) | |
| H15 | 0.4638 | −0.2041 | 0.9117 | 0.061* | |
| H16 | 0.4247 | −0.2273 | 1.0197 | 0.061* | |
| O6W | 0.5887 (4) | 0.2126 (4) | 0.7969 (7) | 0.085 (2) | |
| H17 | 0.6117 | 0.2483 | 0.7682 | 0.127* | |
| H18 | 0.6059 | 0.1919 | 0.8106 | 0.127* | |
| O7W | 0.8981 (4) | −0.0019 (4) | 0.9461 (11) | 0.058 (3) | 0.60 |
| H19 | 0.8703 | 0.0043 | 0.9172 | 0.086* | 0.60 |
| H20 | 0.9281 | 0.0284 | 0.9818 | 0.086* | 0.60 |
| O8W | 0.3286 (5) | −0.2430 (5) | 0.8135 (10) | 0.046 (3) | 0.50 |
| H21 | 0.3628 | −0.2401 | 0.8281 | 0.069* | 0.50 |
| H22 | 0.2989 | −0.2723 | 0.8528 | 0.069* | 0.50 |
| O9W | 0.8626 (7) | −0.0465 (7) | 1.1997 (15) | 0.056 (4) | 0.40 |
| H23 | 0.8442 | −0.0845 | 1.1867 | 0.084* | 0.40 |
| H24 | 0.8733 | −0.0333 | 1.1214 | 0.084* | 0.40 |
| O10W | 0.6667 | 0.3333 | 0.6747 (12) | 0.058 (4) | 0.75 |
| O11W | 0.0224 (9) | −0.0463 (9) | 0.403 (2) | 0.041 (5) | 0.25 |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Nd1 | 0.01785 (17) | 0.00708 (15) | 0.00477 (18) | 0.00439 (11) | −0.00048 (8) | 0.00019 (8) |
| O1 | 0.026 (2) | 0.0124 (17) | 0.0108 (18) | 0.0059 (15) | −0.0037 (15) | 0.0012 (13) |
| O2 | 0.025 (2) | 0.0203 (19) | 0.0088 (17) | 0.0144 (16) | −0.0007 (14) | 0.0038 (14) |
| O3 | 0.0186 (18) | 0.0156 (17) | 0.0116 (18) | 0.0095 (15) | −0.0020 (14) | 0.0033 (14) |
| O4 | 0.025 (2) | 0.0221 (19) | 0.0144 (19) | 0.0157 (17) | 0.0053 (15) | 0.0114 (15) |
| O5 | 0.024 (2) | 0.0142 (17) | 0.0122 (18) | 0.0089 (16) | −0.0010 (14) | −0.0033 (14) |
| O6 | 0.039 (2) | 0.0194 (19) | 0.0120 (19) | 0.0179 (19) | −0.0076 (17) | −0.0075 (15) |
| O7 | 0.025 (2) | 0.0135 (17) | 0.0108 (18) | 0.0096 (15) | −0.0037 (14) | −0.0049 (14) |
| O8 | 0.054 (3) | 0.027 (2) | 0.020 (2) | 0.028 (2) | −0.024 (2) | −0.0138 (17) |
| O9 | 0.025 (2) | 0.0139 (18) | 0.0133 (18) | 0.0089 (16) | 0.0024 (15) | 0.0012 (14) |
| O10 | 0.032 (2) | 0.0157 (19) | 0.0132 (19) | 0.0046 (17) | 0.0092 (16) | −0.0001 (15) |
| O11 | 0.0204 (18) | 0.0142 (17) | 0.0110 (18) | 0.0064 (15) | 0.0037 (14) | 0.0014 (13) |
| O12 | 0.032 (2) | 0.018 (2) | 0.024 (2) | 0.0018 (18) | 0.0131 (18) | −0.0075 (17) |
| N1 | 0.019 (2) | 0.0097 (18) | 0.0068 (19) | 0.0084 (16) | 0.0011 (15) | 0.0001 (15) |
| N2 | 0.022 (2) | 0.0080 (18) | 0.009 (2) | 0.0064 (17) | −0.0020 (16) | −0.0013 (15) |
| N3 | 0.016 (2) | 0.0105 (19) | 0.008 (2) | 0.0038 (16) | −0.0018 (15) | −0.0013 (15) |
| C1 | 0.024 (3) | 0.013 (2) | 0.005 (2) | 0.011 (2) | 0.0002 (18) | −0.0009 (17) |
| C2 | 0.019 (2) | 0.019 (2) | 0.009 (2) | 0.010 (2) | −0.0007 (19) | 0.0010 (19) |
| C3 | 0.018 (3) | 0.023 (3) | 0.015 (3) | 0.008 (2) | −0.001 (2) | 0.004 (2) |
| C4 | 0.021 (3) | 0.016 (2) | 0.015 (3) | 0.010 (2) | 0.001 (2) | 0.0059 (19) |
| C5 | 0.022 (3) | 0.011 (2) | 0.009 (2) | 0.011 (2) | 0.0009 (18) | 0.0000 (17) |
| C6 | 0.025 (3) | 0.012 (2) | 0.006 (2) | 0.009 (2) | 0.0001 (19) | 0.0001 (17) |
| C7 | 0.027 (3) | 0.015 (2) | 0.003 (2) | 0.016 (2) | 0.0022 (18) | 0.0025 (17) |
| C8 | 0.027 (3) | 0.011 (2) | 0.007 (2) | 0.007 (2) | −0.0030 (19) | −0.0007 (18) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C9 | 0.035 (3) | 0.027 (3) | 0.014 (3) | 0.020 (3) | −0.003 (2) | −0.009 (2) |
| C10 | 0.035 (3) | 0.031 (3) | 0.021 (3) | 0.022 (3) | −0.006 (2) | −0.009 (2) |
| C11 | 0.029 (3) | 0.026 (3) | 0.022 (3) | 0.017 (3) | −0.010 (2) | −0.008 (2) |
| C12 | 0.026 (3) | 0.011 (2) | 0.010 (2) | 0.008 (2) | −0.001 (2) | −0.0009 (18) |
| C13 | 0.029 (3) | 0.008 (2) | 0.009 (2) | 0.008 (2) | −0.002 (2) | −0.0015 (17) |
| C14 | 0.035 (3) | 0.012 (2) | 0.007 (2) | 0.010 (2) | −0.005 (2) | −0.0012 (18) |
| C15 | 0.013 (2) | 0.014 (2) | 0.013 (2) | 0.0029 (19) | −0.0017 (18) | −0.0048 (18) |
| C16 | 0.023 (3) | 0.012 (2) | 0.021 (3) | 0.004 (2) | 0.003 (2) | −0.006 (2) |
| C17 | 0.029 (3) | 0.009 (2) | 0.025 (3) | 0.003 (2) | 0.002 (2) | −0.003 (2) |
| C18 | 0.024 (3) | 0.012 (2) | 0.015 (3) | 0.000 (2) | 0.005 (2) | 0.0006 (19) |
| C19 | 0.016 (2) | 0.011 (2) | 0.010 (2) | 0.0024 (19) | −0.0008 (18) | −0.0023 (18) |
| C20 | 0.017 (2) | 0.015 (2) | 0.009 (2) | 0.002 (2) | −0.0007 (19) | −0.0041 (19) |
| C21 | 0.019 (2) | 0.014 (2) | 0.006 (2) | 0.005 (2) | 0.0010 (18) | −0.0004 (18) |
| N2S | 0.031 (3) | 0.023 (2) | 0.012 (2) | 0.022 (2) | −0.0039 (18) | −0.0012 (17) |
| N3S | 0.029 (3) | 0.029 (3) | 0.013 (2) | 0.023 (2) | −0.0060 (18) | 0.0002 (18) |
| C3S | 0.024 (3) | 0.021 (3) | 0.015 (3) | 0.015 (2) | −0.004 (2) | 0.000 (2) |
| C4S | 0.028 (3) | 0.023 (3) | 0.015 (3) | 0.019 (2) | −0.005 (2) | 0.000 (2) |
| C5S | 0.027 (3) | 0.028 (3) | 0.015 (3) | 0.015 (3) | −0.007 (2) | 0.001 (2) |
| C6S | 0.032 (3) | 0.017 (3) | 0.011 (2) | 0.015 (2) | −0.005 (2) | 0.0008 (19) |
| N1S | 0.021 (2) | 0.021 (2) | 0.010 (2) | 0.0104 (19) | −0.0030 (17) | 0.0002 (17) |
| C1S | 0.021 (3) | 0.014 (2) | 0.012 (2) | 0.005 (2) | −0.0055 (19) | −0.0031 (18) |
| C2S | 0.025 (3) | 0.015 (2) | 0.011 (2) | 0.008 (2) | −0.003 (2) | −0.0010 (19) |
| O1W | 0.050 (3) | 0.074 (4) | 0.023 (2) | 0.047 (3) | 0.001 (2) | 0.008 (2) |
| O2W | 0.055 (3) | 0.063 (4) | 0.015 (3) | 0.042 (3) | 0.0003 (19) | 0.0022 (19) |
| O3W | 0.071 (4) | 0.084 (4) | 0.018 (2) | 0.063 (4) | 0.000 (2) | 0.002 (3) |
| O4W | 0.064 (4) | 0.115 (6) | 0.027 (3) | 0.072 (4) | −0.004 (3) | −0.002 (3) |
| O5W | 0.039 (3) | 0.031 (3) | 0.042 (3) | 0.011 (2) | 0.017 (2) | 0.009 (2) |
| O6W | 0.103 (6) | 0.109 (7) | 0.049 (4) | 0.058 (5) | 0.001 (4) | −0.016 (4) |
| O7W | 0.042 (5) | 0.038 (5) | 0.091 (8) | 0.020 (4) | −0.022 (5) | 0.005 (5) |
| O8W | 0.030 (4) | 0.049 (4) | 0.043 (4) | 0.007 (3) | −0.001 (3) | 0.021 (3) |
| O9W | 0.064 (6) | 0.063 (6) | 0.045 (5) | 0.034 (4) | −0.014 (4) | 0.003 (4) |
| O10W | 0.076 (6) | 0.076 (6) | 0.022 (6) | 0.038 (3) | 0.000 | 0.000 |
| O11W | 0.041 (6) | 0.041 (6) | 0.042 (6) | 0.023 (5) | 0.001 (4) | −0.002 (4) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Nd1—O3 | 2.465 (4) | C17—H17A | 0.9500 |
| Nd1—O7 | 2.467 (4) | C18—C19 | 1.390 (7) |
| Nd1—O5 | 2.483 (4) | C18—H18A | 0.9500 |
| Nd1—O1 | 2.482 (4) | C19—C21 | 1.524 (7) |
| Nd1—O9 | 2.485 (4) | N2S—C3S | 1.487 (7) |
| Nd1—O11 | 2.487 (4) | N2S—C6S | 1.501 (8) |
| Nd1—N2 | 2.565 (4) | N2S—H3 | 0.9200 |
| Nd1—N1 | 2.568 (4) | N2S—H4 | 0.9200 |
| Nd1—N3 | 2.575 (4) | N3S—C5S | 1.495 (8) |
| O1—C6 | 1.258 (7) | N3S—C4S | 1.501 (8) |
| O2—C6 | 1.246 (6) | N3S—H5 | 0.9200 |
| O3—C7 | 1.247 (7) | N3S—H6 | 0.9200 |
| O4—C7 | 1.255 (6) | C3S—C4S | 1.521 (7) |

| | | | |
|---|---|---|---|
| O5—C13 | 1.278 (7) | C3S—H3SB | 0.9900 |
| O6—C13 | 1.235 (6) | C3S—H3SC | 0.9900 |
| O7—C14 | 1.240 (7) | C4S—H4SA | 0.9900 |
| O8—C14 | 1.254 (7) | C4S—H4SB | 0.9900 |
| O9—C21 | 1.263 (6) | C5S—C6S | 1.503 (8) |
| O10—C21 | 1.236 (6) | C5S—H5SA | 0.9900 |
| O11—C20 | 1.254 (6) | C5S—H5SB | 0.9900 |
| O12—C20 | 1.245 (7) | C6S—H6SA | 0.9900 |
| N1—C1 | 1.333 (6) | C6S—H6SB | 0.9900 |
| N1—C5 | 1.340 (6) | N1S—C2S | 1.497 (7) |
| N2—C8 | 1.338 (6) | N1S—C1S | 1.499 (7) |
| N2—C12 | 1.341 (7) | N1S—H1 | 0.9200 |
| N3—C19 | 1.332 (7) | N1S—H2 | 0.9200 |
| N3—C15 | 1.340 (7) | C1S—C2S$^{i}$ | 1.515 (8) |
| C1—C2 | 1.386 (7) | C1S—H1SB | 0.9900 |
| C1—C6 | 1.524 (7) | C1S—H1SC | 0.9900 |
| C2—C3 | 1.401 (7) | C2S—C1S$^{i}$ | 1.515 (8) |
| C2—H2A | 0.9500 | C2S—H2SB | 0.9900 |
| C3—C4 | 1.393 (7) | C2S—H2SC | 0.9900 |
| C3—H3A | 0.9500 | O1W—H7 | 0.8499 |
| C4—C5 | 1.386 (7) | O1W—H8 | 0.8500 |
| C4—H4A | 0.9500 | O2W—H9 | 0.8501 |
| C5—C7 | 1.516 (7) | O2W—H10 | 0.8498 |
| C7—O4 | 1.255 (6) | O3W—H11 | 0.8500 |
| C8—C9 | 1.375 (8) | O3W—H12 | 0.8501 |
| C8—C13 | 1.521 (7) | O4W—H13 | 0.8500 |
| C9—C10 | 1.401 (8) | O4W—H14 | 0.8498 |
| C9—H9A | 0.9500 | O5W—H15 | 0.8500 |
| C10—C11 | 1.383 (8) | O5W—H16 | 0.8502 |
| C10—H10A | 0.9500 | O6W—H17 | 0.8499 |
| C11—C12 | 1.374 (8) | O6W—H18 | 0.8500 |
| C11—H11A | 0.9500 | O7W—H19 | 0.8503 |
| C12—C14 | 1.523 (7) | O7W—H20 | 0.8494 |
| C15—C16 | 1.393 (7) | O8W—H21 | 0.8501 |
| C15—C20 | 1.517 (7) | O8W—H22 | 0.8500 |
| C16—C17 | 1.390 (8) | O9W—H23 | 0.8500 |
| C16—H16A | 0.9500 | O9W—H24 | 0.8499 |
| C17—C18 | 1.398 (8) | | |
| O3—Nd1—O7 | 77.20 (13) | O6—C13—C8 | 118.2 (5) |
| O3—Nd1—O5 | 90.11 (12) | O5—C13—C8 | 115.6 (4) |
| O7—Nd1—O5 | 125.79 (12) | O7—C14—O8 | 125.4 (5) |
| O3—Nd1—O1 | 125.64 (12) | O7—C14—C12 | 117.1 (5) |
| O7—Nd1—O1 | 151.47 (12) | O8—C14—C12 | 117.6 (5) |
| O5—Nd1—O1 | 75.51 (13) | N3—C15—C16 | 121.8 (5) |
| O3—Nd1—O9 | 150.59 (12) | N3—C15—C20 | 114.5 (4) |
| O7—Nd1—O9 | 88.93 (13) | C16—C15—C20 | 123.6 (5) |
| O5—Nd1—O9 | 77.05 (13) | C17—C16—C15 | 118.3 (5) |
| O1—Nd1—O9 | 77.04 (13) | C17—C16—H16A | 120.8 |

| | | | |
|---|---|---|---|
| O3—Nd1—O11 | 77.45 (12) | C15—C16—H16A | 120.8 |
| O7—Nd1—O11 | 78.02 (13) | C16—C17—C18 | 119.8 (5) |
| O5—Nd1—O11 | 150.31 (13) | C16—C17—H17A | 120.1 |
| O1—Nd1—O11 | 89.88 (13) | C18—C17—H17A | 120.1 |
| O9—Nd1—O11 | 125.28 (12) | C19—C18—C17 | 117.8 (5) |
| O3—Nd1—N2 | 77.80 (13) | C19—C18—H18A | 121.1 |
| O7—Nd1—N2 | 62.76 (13) | C17—C18—H18A | 121.1 |
| O5—Nd1—N2 | 63.06 (13) | N3—C19—C18 | 122.6 (5) |
| O1—Nd1—N2 | 132.85 (13) | N3—C19—C21 | 115.4 (4) |
| O9—Nd1—N2 | 72.79 (13) | C18—C19—C21 | 122.0 (5) |
| O11—Nd1—N2 | 137.21 (13) | O12—C20—O11 | 125.4 (5) |
| O3—Nd1—N1 | 62.48 (12) | O12—C20—C15 | 118.5 (5) |
| O7—Nd1—N1 | 136.93 (13) | O11—C20—C15 | 116.0 (5) |
| O5—Nd1—N1 | 71.35 (13) | O10—C21—O9 | 126.3 (5) |
| O1—Nd1—N1 | 63.23 (13) | O10—C21—C19 | 118.2 (5) |
| O9—Nd1—N1 | 133.88 (13) | O9—C21—C19 | 115.5 (4) |
| O11—Nd1—N1 | 79.02 (13) | C3S—N2S—C6S | 112.1 (4) |
| N2—Nd1—N1 | 118.22 (12) | C3S—N2S—H3 | 109.2 |
| O3—Nd1—N3 | 136.85 (13) | C6S—N2S—H3 | 109.2 |
| O7—Nd1—N3 | 79.27 (13) | C3S—N2S—H4 | 109.2 |
| O5—Nd1—N3 | 132.81 (13) | C6S—N2S—H4 | 109.2 |
| O1—Nd1—N3 | 72.23 (13) | H3—N2S—H4 | 107.9 |
| O9—Nd1—N3 | 62.87 (13) | C5S—N3S—C4S | 111.7 (4) |
| O11—Nd1—N3 | 62.53 (13) | C5S—N3S—H5 | 109.3 |
| N2—Nd1—N3 | 121.23 (12) | C4S—N3S—H5 | 109.3 |
| N1—Nd1—N3 | 120.15 (12) | C5S—N3S—H6 | 109.3 |
| C6—O1—Nd1 | 124.6 (3) | C4S—N3S—H6 | 109.3 |
| C7—O3—Nd1 | 126.3 (3) | H5—N3S—H6 | 107.9 |
| C13—O5—Nd1 | 125.3 (3) | N2S—C3S—C4S | 110.3 (4) |
| C14—O7—Nd1 | 125.8 (3) | N2S—C3S—H3SB | 109.6 |
| C21—O9—Nd1 | 125.4 (3) | C4S—C3S—H3SB | 109.6 |
| C20—O11—Nd1 | 126.3 (3) | N2S—C3S—H3SC | 109.6 |
| C1—N1—C5 | 119.2 (5) | C4S—C3S—H3SC | 109.6 |
| C1—N1—Nd1 | 119.5 (3) | H3SB—C3S—H3SC | 108.1 |
| C5—N1—Nd1 | 120.7 (3) | N3S—C4S—C3S | 109.9 (4) |
| C8—N2—C12 | 118.8 (5) | N3S—C4S—H4SA | 109.7 |
| C8—N2—Nd1 | 120.3 (3) | C3S—C4S—H4SA | 109.7 |
| C12—N2—Nd1 | 120.7 (3) | N3S—C4S—H4SB | 109.7 |
| C19—N3—C15 | 119.7 (4) | C3S—C4S—H4SB | 109.7 |
| C19—N3—Nd1 | 119.6 (3) | H4SA—C4S—H4SB | 108.2 |
| C15—N3—Nd1 | 120.4 (3) | N3S—C5S—C6S | 110.8 (5) |
| N1—C1—C2 | 122.3 (5) | N3S—C5S—H5SA | 109.5 |
| N1—C1—C6 | 114.9 (5) | C6S—C5S—H5SA | 109.5 |
| C2—C1—C6 | 122.8 (5) | N3S—C5S—H5SB | 109.5 |
| C1—C2—C3 | 118.7 (5) | C6S—C5S—H5SB | 109.5 |
| C1—C2—H2A | 120.6 | H5SA—C5S—H5SB | 108.1 |
| C3—C2—H2A | 120.6 | C5S—C6S—N2S | 109.9 (4) |
| C4—C3—C2 | 118.7 (5) | C5S—C6S—H6SA | 109.7 |
| C4—C3—H3A | 120.6 | N2S—C6S—H6SA | 109.7 |

| | | | |
|---|---|---|---|
| C2—C3—H3A | 120.6 | C5S—C6S—H6SB | 109.7 |
| C5—C4—C3 | 118.5 (5) | N2S—C6S—H6SB | 109.7 |
| C5—C4—H4A | 120.8 | H6SA—C6S—H6SB | 108.2 |
| C3—C4—H4A | 120.8 | C2S—N1S—C1S | 111.8 (4) |
| N1—C5—C4 | 122.6 (5) | C2S—N1S—H1 | 109.3 |
| N1—C5—C7 | 113.2 (4) | C1S—N1S—H1 | 109.3 |
| C4—C5—C7 | 124.2 (5) | C2S—N1S—H2 | 109.3 |
| O2—C6—O1 | 126.1 (5) | C1S—N1S—H2 | 109.3 |
| O2—C6—C1 | 117.3 (5) | H1—N1S—H2 | 107.9 |
| O1—C6—C1 | 116.6 (4) | N1S—C1S—C2S$^{i}$ | 110.9 (4) |
| O3—C7—O4 | 125.3 (5) | N1S—C1S—H1SB | 109.5 |
| O3—C7—O4 | 125.3 (5) | C2S$^{i}$—C1S—H1SB | 109.5 |
| O3—C7—C5 | 116.6 (4) | N1S—C1S—H1SC | 109.5 |
| O4—C7—C5 | 118.1 (5) | C2S$^{i}$—C1S—H1SC | 109.5 |
| O4—C7—C5 | 118.1 (5) | H1SB—C1S—H1SC | 108.0 |
| N2—C8—C9 | 122.9 (5) | N1S—C2S—C1S$^{i}$ | 110.0 (4) |
| N2—C8—C13 | 115.0 (5) | N1S—C2S—H2SB | 109.7 |
| C9—C8—C13 | 122.1 (5) | C1S$^{i}$—C2S—H2SB | 109.7 |
| C8—C9—C10 | 118.1 (5) | N1S—C2S—H2SC | 109.7 |
| C8—C9—H9A | 120.9 | C1S$^{i}$—C2S—H2SC | 109.7 |
| C10—C9—H9A | 120.9 | H2SB—C2S—H2SC | 108.2 |
| C11—C10—C9 | 118.8 (6) | H7—O1W—H8 | 117.6 |
| C11—C10—H10A | 120.6 | H9—O2W—H10 | 109.6 |
| C9—C10—H10A | 120.6 | H11—O3W—H12 | 118.7 |
| C12—C11—C10 | 119.2 (5) | H13—O4W—H14 | 105.9 |
| C12—C11—H11A | 120.4 | H15—O5W—H16 | 110.1 |
| C10—C11—H11A | 120.4 | H17—O6W—H18 | 115.1 |
| N2—C12—C11 | 122.1 (5) | H19—O7W—H20 | 115.6 |
| N2—C12—C14 | 113.1 (5) | H21—O8W—H22 | 114.8 |
| C11—C12—C14 | 124.8 (5) | H23—O9W—H24 | 101.1 |
| O6—C13—O5 | 126.2 (5) | | |
| O3—Nd1—O1—C6 | 12.5 (5) | N2—Nd1—N3—C15 | 132.0 (4) |
| O7—Nd1—O1—C6 | 150.9 (4) | N1—Nd1—N3—C15 | −55.3 (4) |
| O5—Nd1—O1—C6 | −66.9 (4) | C5—N1—C1—C2 | 1.3 (7) |
| O9—Nd1—O1—C6 | −146.7 (4) | Nd1—N1—C1—C2 | −170.3 (4) |
| O11—Nd1—O1—C6 | 86.9 (4) | C5—N1—C1—C6 | −179.7 (4) |
| N2—Nd1—O1—C6 | −95.5 (4) | Nd1—N1—C1—C6 | 8.7 (5) |
| N1—Nd1—O1—C6 | 9.2 (4) | N1—C1—C2—C3 | −1.0 (8) |
| N3—Nd1—O1—C6 | 148.0 (4) | C6—C1—C2—C3 | −179.8 (5) |
| O7—Nd1—O3—C7 | −160.7 (4) | C1—C2—C3—C4 | −0.5 (8) |
| O5—Nd1—O3—C7 | 72.5 (4) | C2—C3—C4—C5 | 1.5 (8) |
| O1—Nd1—O3—C7 | 0.4 (5) | C1—N1—C5—C4 | −0.2 (7) |
| O9—Nd1—O3—C7 | 135.6 (4) | Nd1—N1—C5—C4 | 171.3 (4) |
| O11—Nd1—O3—C7 | −80.3 (4) | C1—N1—C5—C7 | −178.7 (4) |
| N2—Nd1—O3—C7 | 134.9 (4) | Nd1—N1—C5—C7 | −7.3 (5) |
| N1—Nd1—O3—C7 | 3.7 (4) | C3—C4—C5—N1 | −1.2 (8) |
| N3—Nd1—O3—C7 | −102.1 (4) | C3—C4—C5—C7 | 177.2 (5) |

| | | | |
|---|---|---|---|
| O3—Nd1—O5—C13 | 84.2 (4) | Nd1—O1—C6—O2 | 171.7 (4) |
| O7—Nd1—O5—C13 | 9.9 (5) | Nd1—O1—C6—C1 | −8.4 (6) |
| O1—Nd1—O5—C13 | −148.9 (4) | N1—C1—C6—O2 | 179.2 (4) |
| O9—Nd1—O5—C13 | −69.1 (4) | C2—C1—C6—O2 | −1.8 (7) |
| O11—Nd1—O5—C13 | 148.4 (4) | N1—C1—C6—O1 | −0.7 (7) |
| N2—Nd1—O5—C13 | 7.9 (4) | C2—C1—C6—O1 | 178.3 (5) |
| N1—Nd1—O5—C13 | 144.9 (4) | Nd1—O3—C7—O4 | 171.8 (4) |
| N3—Nd1—O5—C13 | −100.8 (4) | Nd1—O3—C7—O4 | 171.8 (4) |
| O3—Nd1—O7—C14 | −78.3 (5) | Nd1—O3—C7—C5 | −8.8 (6) |
| O5—Nd1—O7—C14 | 2.5 (5) | O4—O4—C7—O3 | 0.00 (15) |
| O1—Nd1—O7—C14 | 135.3 (4) | O4—O4—C7—C5 | 0.0 (3) |
| O9—Nd1—O7—C14 | 75.6 (4) | N1—C5—C7—O3 | 10.1 (6) |
| O11—Nd1—O7—C14 | −157.9 (5) | C4—C5—C7—O3 | −168.4 (5) |
| N2—Nd1—O7—C14 | 4.5 (4) | N1—C5—C7—O4 | −170.4 (4) |
| N1—Nd1—O7—C14 | −98.7 (5) | C4—C5—C7—O4 | 11.1 (7) |
| N3—Nd1—O7—C14 | 138.2 (5) | N1—C5—C7—O4 | −170.4 (4) |
| O3—Nd1—O9—C21 | 149.9 (4) | C4—C5—C7—O4 | 11.1 (7) |
| O7—Nd1—O9—C21 | 88.9 (4) | C12—N2—C8—C9 | 1.7 (8) |
| O5—Nd1—O9—C21 | −143.9 (4) | Nd1—N2—C8—C9 | −172.5 (4) |
| O1—Nd1—O9—C21 | −66.1 (4) | C12—N2—C8—C13 | 179.7 (5) |
| O11—Nd1—O9—C21 | 14.4 (5) | Nd1—N2—C8—C13 | 5.4 (6) |
| N2—Nd1—O9—C21 | 150.6 (4) | N2—C8—C9—C10 | −1.5 (9) |
| N1—Nd1—O9—C21 | −96.5 (4) | C13—C8—C9—C10 | −179.3 (5) |
| N3—Nd1—O9—C21 | 10.4 (4) | C8—C9—C10—C11 | 0.1 (10) |
| O3—Nd1—O11—C20 | −161.4 (5) | C9—C10—C11—C12 | 1.0 (10) |
| O7—Nd1—O11—C20 | −82.1 (5) | C8—N2—C12—C11 | −0.6 (8) |
| O5—Nd1—O11—C20 | 131.3 (4) | Nd1—N2—C12—C11 | 173.7 (4) |
| O1—Nd1—O11—C20 | 71.9 (4) | C8—N2—C12—C14 | −179.4 (5) |
| O9—Nd1—O11—C20 | −2.1 (5) | Nd1—N2—C12—C14 | −5.1 (6) |
| N2—Nd1—O11—C20 | −105.4 (4) | C10—C11—C12—N2 | −0.8 (9) |
| N1—Nd1—O11—C20 | 134.6 (4) | C10—C11—C12—C14 | 177.8 (6) |
| N3—Nd1—O11—C20 | 1.9 (4) | Nd1—O5—C13—O6 | 172.8 (4) |
| O3—Nd1—N1—C1 | 174.1 (4) | Nd1—O5—C13—C8 | −8.2 (6) |
| O7—Nd1—N1—C1 | −163.3 (3) | N2—C8—C13—O6 | −179.5 (5) |
| O5—Nd1—N1—C1 | 73.8 (4) | C9—C8—C13—O6 | −1.5 (8) |
| O1—Nd1—N1—C1 | −9.0 (3) | N2—C8—C13—O5 | 1.4 (7) |
| O9—Nd1—N1—C1 | 24.6 (4) | C9—C8—C13—O5 | 179.4 (5) |
| O11—Nd1—N1—C1 | −104.4 (4) | Nd1—O7—C14—O8 | 171.0 (5) |
| N2—Nd1—N1—C1 | 117.5 (3) | Nd1—O7—C14—C12 | −8.7 (7) |
| N3—Nd1—N1—C1 | −55.5 (4) | N2—C12—C14—O7 | 8.7 (7) |
| O3—Nd1—N1—C5 | 2.7 (3) | C11—C12—C14—O7 | −170.1 (6) |
| O7—Nd1—N1—C5 | 25.3 (4) | N2—C12—C14—O8 | −171.1 (5) |
| O5—Nd1—N1—C5 | −97.6 (4) | C11—C12—C14—O8 | 10.1 (9) |
| O1—Nd1—N1—C5 | 179.6 (4) | C19—N3—C15—C16 | −0.1 (8) |
| O9—Nd1—N1—C5 | −146.8 (3) | Nd1—N3—C15—C16 | 173.9 (4) |
| O11—Nd1—N1—C5 | 84.1 (4) | C19—N3—C15—C20 | −177.7 (5) |
| N2—Nd1—N1—C5 | −53.9 (4) | Nd1—N3—C15—C20 | −3.6 (6) |
| N3—Nd1—N1—C5 | 133.1 (3) | N3—C15—C16—C17 | −1.1 (9) |
| O3—Nd1—N2—C8 | −103.0 (4) | C20—C15—C16—C17 | 176.2 (5) |

| | | | |
|---|---|---|---|
| O7—Nd1—N2—C8 | 175.3 (4) | C15—C16—C17—C18 | 1.1 (9) |
| O5—Nd1—N2—C8 | −6.5 (4) | C16—C17—C18—C19 | 0.0 (9) |
| O1—Nd1—N2—C8 | 24.8 (5) | C15—N3—C19—C18 | 1.4 (8) |
| O9—Nd1—N2—C8 | 77.4 (4) | Nd1—N3—C19—C18 | −172.7 (4) |
| O11—Nd1—N2—C8 | −158.9 (3) | C15—N3—C19—C21 | −179.0 (5) |
| N1—Nd1—N2—C8 | −53.7 (4) | Nd1—N3—C19—C21 | 6.9 (6) |
| N3—Nd1—N2—C8 | 119.1 (4) | C17—C18—C19—N3 | −1.3 (9) |
| O3—Nd1—N2—C12 | 82.9 (4) | C17—C18—C19—C21 | 179.1 (5) |
| O7—Nd1—N2—C12 | 1.1 (4) | Nd1—O11—C20—O12 | 175.9 (4) |
| O5—Nd1—N2—C12 | 179.3 (4) | Nd1—O11—C20—C15 | −4.4 (7) |
| O1—Nd1—N2—C12 | −149.4 (4) | N3—C15—C20—O12 | −175.2 (5) |
| O9—Nd1—N2—C12 | −96.8 (4) | C16—C15—C20—O12 | 7.3 (8) |
| O11—Nd1—N2—C12 | 27.0 (5) | N3—C15—C20—O11 | 5.1 (7) |
| N1—Nd1—N2—C12 | 132.1 (4) | C16—C15—C20—O11 | −172.4 (5) |
| N3—Nd1—N2—C12 | −55.0 (4) | Nd1—O9—C21—O10 | 168.0 (4) |
| O3—Nd1—N3—C19 | −160.6 (3) | Nd1—O9—C21—C19 | −10.9 (6) |
| O7—Nd1—N3—C19 | −102.7 (4) | N3—C19—C21—O10 | −177.0 (5) |
| O5—Nd1—N3—C19 | 26.8 (5) | C18—C19—C21—O10 | 2.7 (8) |
| O1—Nd1—N3—C19 | 75.9 (4) | N3—C19—C21—O9 | 2.0 (7) |
| O9—Nd1—N3—C19 | −8.4 (4) | C18—C19—C21—O9 | −178.4 (5) |
| O11—Nd1—N3—C19 | 175.3 (4) | C6S—N2S—C3S—C4S | −57.2 (6) |
| N2—Nd1—N3—C19 | −54.0 (4) | C5S—N3S—C4S—C3S | −56.4 (6) |
| N1—Nd1—N3—C19 | 118.7 (4) | N2S—C3S—C4S—N3S | 55.9 (6) |
| O3—Nd1—N3—C15 | 25.4 (5) | C4S—N3S—C5S—C6S | 57.1 (6) |
| O7—Nd1—N3—C15 | 83.2 (4) | N3S—C5S—C6S—N2S | −56.2 (6) |
| O5—Nd1—N3—C15 | −147.3 (4) | C3S—N2S—C6S—C5S | 57.2 (6) |
| O1—Nd1—N3—C15 | −98.2 (4) | C2S—N1S—C1S—C2S$^{i}$ | −56.8 (6) |
| O9—Nd1—N3—C15 | 177.6 (4) | C1S—N1S—C2S—C1S$^{i}$ | 56.3 (6) |
| O11—Nd1—N3—C15 | 1.3 (4) | | |

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

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2S—H3···O4 | 0.92 | 1.97 | 2.804 (6) | 151 |
| N2S—H4···O6$^{ii}$ | 0.92 | 1.92 | 2.760 (6) | 151 |
| N3S—H5···O10$^{iii}$ | 0.92 | 1.95 | 2.780 (6) | 150 |
| N3S—H6···O8$^{iv}$ | 0.92 | 1.99 | 2.836 (6) | 153 |
| N1S—H1···O2$^{v}$ | 0.92 | 1.92 | 2.758 (6) | 150 |
| N1S—H2···O12$^{vi}$ | 0.92 | 2.03 | 2.874 (6) | 153 |
| O1W—H7···O9$^{iii}$ | 0.85 | 2.03 | 2.863 (7) | 167 |
| O1W—H8···O9W$^{vii}$ | 0.85 | 2.01 | 2.838 (17) | 167 |
| O2W—H9···O1$^{v}$ | 0.85 | 2.43 | 3.278 (7) | 180 |
| O2W—H10···O1W$^{iii}$ | 0.85 | 2.17 | 3.024 (8) | 180 |
| O3W—H11···O5$^{ii}$ | 0.85 | 2.14 | 2.970 (7) | 167 |
| O3W—H12···O6W$^{ii}$ | 0.85 | 2.11 | 2.950 (11) | 171 |

| | | | | |
|---|---|---|---|---|
| O4W—H13···O1$^{v}$ | 0.85 | 2.05 | 2.878 (7) | 166 |
| O4W—H14···O8W$^{v}$ | 0.85 | 2.17 | 2.985 (14) | 162 |
| O5W—H15···O4$^{viii}$ | 0.85 | 1.82 | 2.674 (6) | 180 |
| O5W—H16···O6W$^{ix}$ | 0.85 | 1.95 | 2.804 (9) | 179 |
| O6W—H17···O10W | 0.85 | 2.13 | 2.972 (10) | 174 |
| O6W—H18···O12$^{viii}$ | 0.85 | 2.16 | 3.011 (10) | 180 |
| O7W—H19···O8$^{viii}$ | 0.85 | 1.76 | 2.608 (10) | 179 |
| O7W—H20···O7W$^{x}$ | 0.85 | 1.95 | 2.797 (11) | 177 |
| O8W—H21···O5W | 0.85 | 1.99 | 2.787 (12) | 156 |
| O8W—H22···O5W$^{xi}$ | 0.85 | 1.87 | 2.723 (11) | 180 |
| O9W—H23···O8$^{xii}$ | 0.85 | 2.29 | 3.136 (18) | 179 |
| O9W—H24···O7W | 0.85 | 1.92 | 2.765 (20) | 179 |
| C3S—H3Sc···O10$^{iii}$ | 0.99 | 2.49 | 3.200 (8) | 129 |
| C4S—H4SB···O6$^{ii}$ | 0.99 | 2.47 | 3.179 (8) | 128 |
| C4S—H4SB···O7$^{iv}$ | 0.99 | 2.51 | 3.076 (9) | 116 |
| C5S—H5SA···O6$^{ii}$ | 0.99 | 2.46 | 3.180 (8) | 129 |
| C6S—H6SB···O3 | 0.99 | 2.52 | 3.092 (8) | 116 |
| C6S—H6SB···O10$^{iii}$ | 0.99 | 2.45 | 3.169 (7) | 129 |
| C1S—H1SC···O2$^{xiii}$ | 0.99 | 2.43 | 3.151 (7) | 129 |
| C2S—H2SB···O11$^{vi}$ | 0.99 | 2.55 | 3.112 (8) | 116 |
| C2S—H2SB···O2$^{xiii}$ | 0.99 | 2.49 | 3.194 (7) | 128 |

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

**Fig. 1**

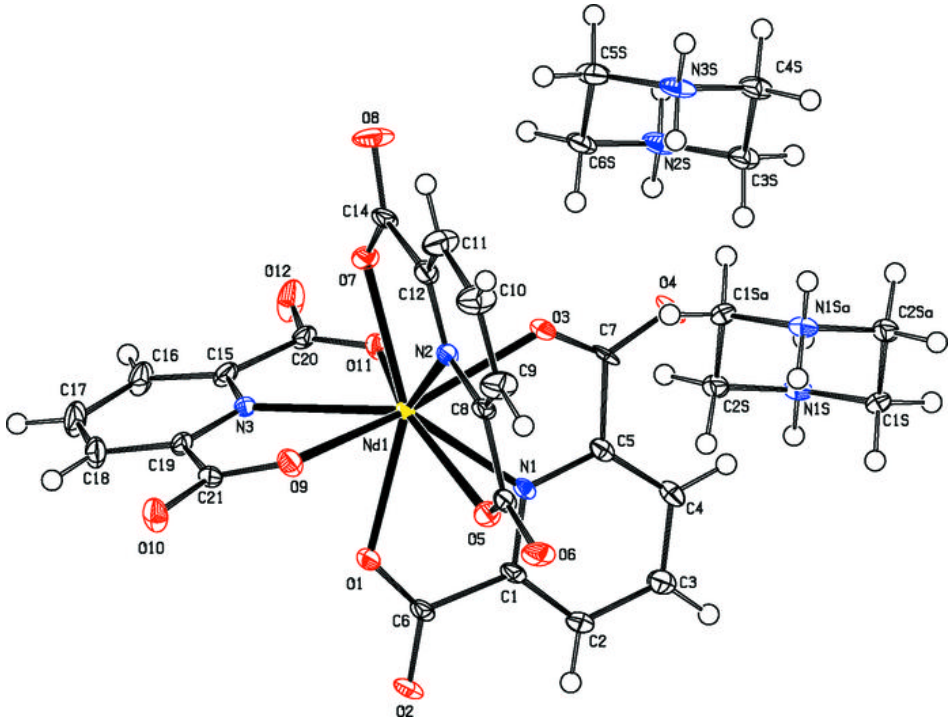
C5S
N3S
C4S
C6S
N2S
C3S
O8
C14
C11
O12
O7
C12
C10
O4
C1Sa
N1Sa
C2Sa
O3
C7
C16
C15
C20
O11
N2
C9
C17
C8
C2S
N1S
C1S
N3
Nd1
C5
C19
C18
N1
C21
O9
C4
O10
O5
O6
O1
C3
C1
C6
C2
O2

**Fig. 2**

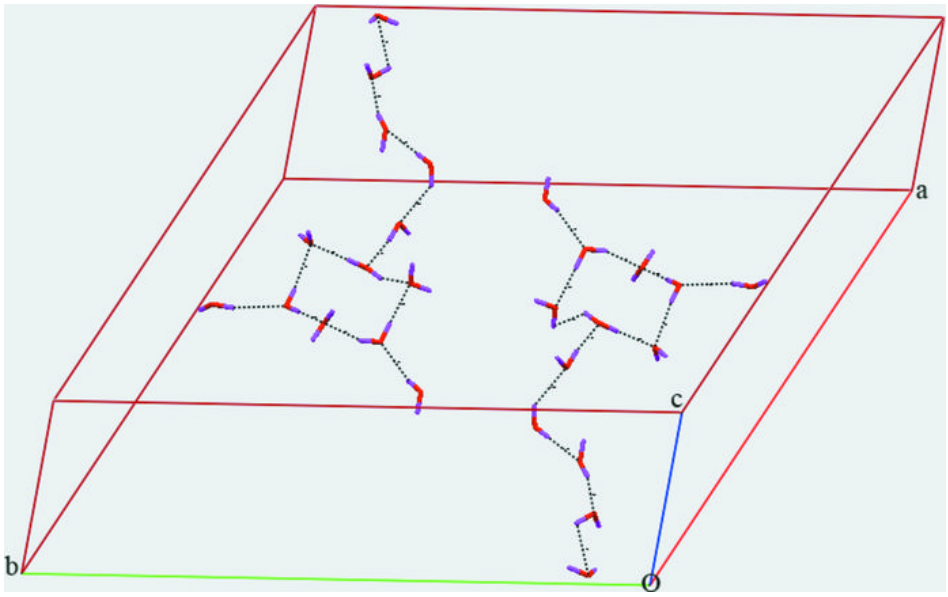

**Fig. 3**

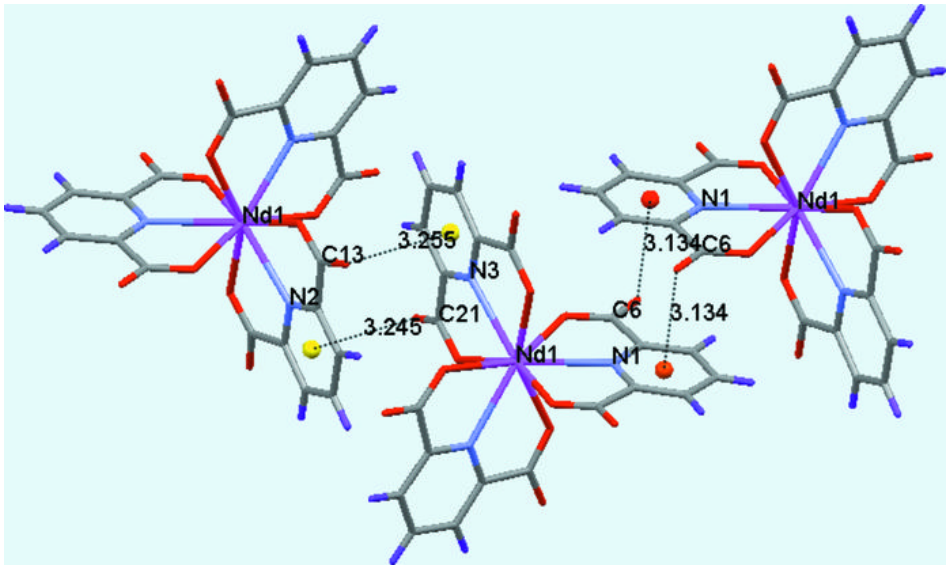

**Fig. 4**