

# catena-Poly[diimidazolium [bis( $\mu$ -pyridine-2,5-dicarboxylato)bis[diaqua-praseodymate(III)]]-bis( $\mu$ -pyridine-2,5-dicarboxylato)]

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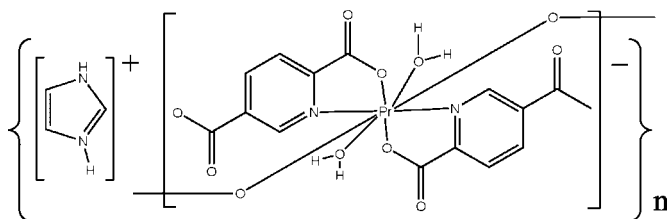
Received 24 March 2011; accepted 3 April 2011

Key indicators: single-crystal X-ray study;  $T = 293$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.015;  $wR$  factor = 0.043; data-to-parameter ratio = 11.7.

The title compound  $\{(\text{C}_3\text{H}_5\text{N}_2)_2[\text{Pr}_2(\text{C}_7\text{H}_3\text{NO}_4)_4(\text{H}_2\text{O})_4]\}_n$ , has a chain structure featuring a dimeric unit consisting of two  $\text{Pr}^{\text{III}}$  atoms within a dodecahedral environment. Each of the metal cations is coordinated by two N atoms and two O atoms from two pyridine-2,5-dicarboxylate ligands, two O atoms from another two pyridine-2,5-dicarboxylate ligands and two water O atoms. The  $\text{Pr}^{\text{III}}$  ions are bridged by two ligands along the  $c$  axis, forming the dimeric unit, and these are connected by four ligands along the  $b$  axis, forming a chain.  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds are found in the structure.

## Related literature

For praseodymium complexes with pyridine-dicarboxylate ligands, see: Chen *et al.* (2011); Zhao *et al.* (2009); Song *et al.* (2006); Chi *et al.* (2009). For complexes with similar structures, see: Li, Zhang *et al.* (2009); Li, Chen *et al.* (2009); Huang *et al.* (2009); Zhang *et al.* (2005, 2007).



## Experimental

### Crystal data

$(\text{C}_3\text{H}_5\text{N}_2)_2[\text{Pr}_2(\text{C}_7\text{H}_3\text{NO}_4)_4(\text{H}_2\text{O})_4]$   
 $M_r = 1152.48$

Triclinic,  $P\bar{1}$   
 $a = 9.5444$  (19) Å

$b = 10.667$  (2) Å  
 $c = 11.222$  (2) Å  
 $\alpha = 64.63$  (3)°  
 $\beta = 79.50$  (3)°  
 $\gamma = 87.50$  (3)°  
 $V = 1014.3$  (3) Å<sup>3</sup>

$Z = 1$   
Mo  $K\alpha$  radiation  
 $\mu = 2.47$  mm<sup>-1</sup>  
 $T = 293$  K  
 $0.19 \times 0.16 \times 0.09$  mm

### Data collection

Bruker SMART CCD area-detector diffractometer  
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)  
 $T_{\min} = 0.652$ ,  $T_{\max} = 0.809$

14917 measured reflections  
3561 independent reflections  
3468 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.020$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.015$   
 $wR(F^2) = 0.043$   
 $S = 1.04$   
3561 reflections  
305 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 0.33$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.45$  e Å<sup>-3</sup>

Table 1

Selected geometric parameters (Å, °).

|                      |             |         |             |
|----------------------|-------------|---------|-------------|
| Pr1—O5               | 2.3835 (17) | Pr1—O10 | 2.4643 (18) |
| Pr1—O4 <sup>i</sup>  | 2.4193 (16) | Pr1—O9  | 2.459 (2)   |
| Pr1—O7 <sup>ii</sup> | 2.4366 (16) | Pr1—N1  | 2.6484 (18) |
| Pr1—O1               | 2.4407 (15) | Pr1—N2  | 2.677 (2)   |

Symmetry codes: (i)  $x - 1, y, z$ ; (ii)  $-x + 1, -y, -z + 1$ .

Table 2

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$                                | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|-----------------------------------------------------|--------------|--------------------|-------------|----------------------|
| $\text{N4}-\text{H4}\cdots\text{O2}^{\text{iv}}$    | 0.86         | 1.85               | 2.679 (3)   | 160                  |
| $\text{N3}-\text{H3A}\cdots\text{O6}^{\text{v}}$    | 0.86         | 1.88               | 2.735 (3)   | 172                  |
| $\text{N3}-\text{H3A}\cdots\text{O5}^{\text{v}}$    | 0.86         | 2.59               | 3.063 (3)   | 115                  |
| $\text{O9}-\text{H9A}\cdots\text{O1}^{\text{vi}}$   | 0.77 (3)     | 1.98 (3)           | 2.743 (3)   | 172 (3)              |
| $\text{O10}-\text{H10A}\cdots\text{O3}^{\text{i}}$  | 0.88 (4)     | 1.77 (4)           | 2.645 (3)   | 173 (3)              |
| $\text{O10}-\text{H10A}\cdots\text{O4}^{\text{i}}$  | 0.88 (4)     | 2.44 (3)           | 2.894 (3)   | 113 (3)              |
| $\text{O10}-\text{H10B}\cdots\text{O3}^{\text{ii}}$ | 0.73 (4)     | 2.10 (4)           | 2.789 (3)   | 157 (4)              |
| $\text{O9}-\text{H9B}\cdots\text{O8}^{\text{ii}}$   | 0.73 (3)     | 1.96 (4)           | 2.644 (3)   | 157 (3)              |

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); 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: JH2277).

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## supporting information

*Acta Cryst.* (2011). E67, m569–m570 [https://doi.org/10.1107/S1600536811012360]

**catena-Poly[diimidazolium [bis( $\mu$ -pyridine-2,5-dicarboxylato)bis[di aqua-praseodymate(III)]]-bis( $\mu$ -pyridine-2,5-dicarboxylato)]**

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### S1. Comment

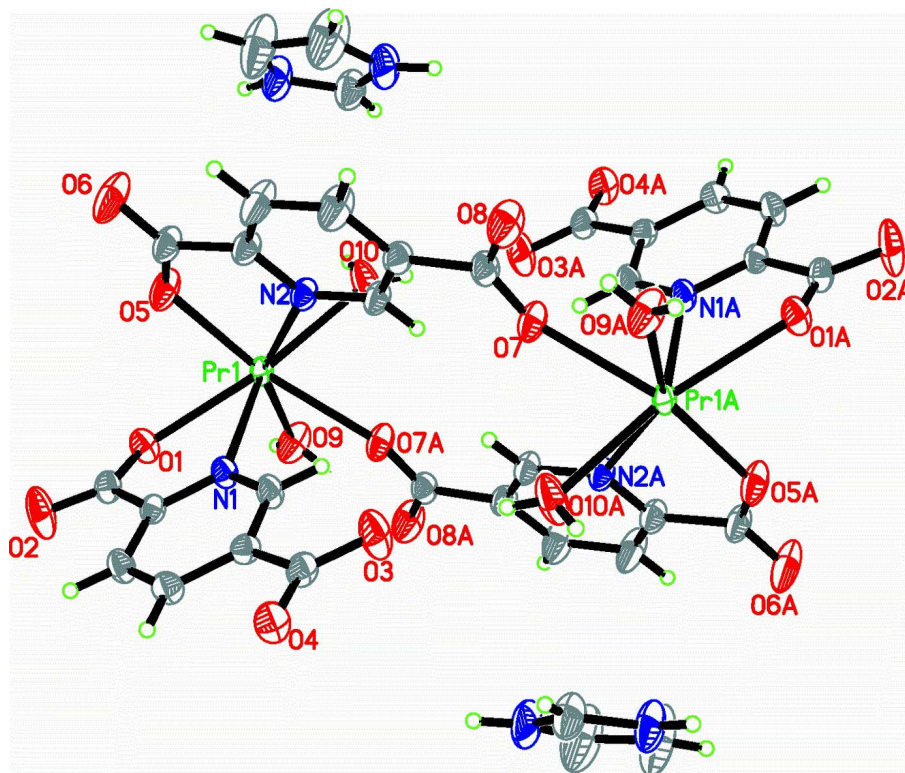
Pyridine-dicarboxylic acids as multidentate ligands containing N- and O- donors, have been widely used in preparing many kinds of coordination complexes especially in complexes containing rare earth metals (Chen *et al.* 2011, Zhao *et al.* 2009). Many complexes based on pyridine-2,5-dicarboxylic acid have been prepared (Li, Zhang *et al.* 2009, Li, Chen *et al.* 2009, Huang *et al.* 2009, Zhang *et al.* 2007, Zhang *et al.* 2005), but no complex containing Pr element, except two 3 d-4f complexes (Song *et al.* 2006, Chi *et al.* 2009) was reported. The reaction of pyridine-2,5-dicarboxylic acid with praseodymium salt under hydrothermal conditions results in the formation of a complex formulated as  $\{(\text{C}_3\text{N}_2\text{H}_5)[(\text{C}_7\text{H}_3\text{NO}_4)_2\text{Pr}(\text{H}_2\text{O})_2]\}_n$ , and the complex has been structurally characterized by elemental analysis and X-ray diffraction. The structure of **1** viewed down the *b*-axis was presented in Fig. 2. Hydrogen bonding packing diagram of **1** view down the *b*-axis was shown in Fig. 3. Selected bond lengths and angles were summarized in Table 1. The distances of the hydrogen bands were listed in Table 2.

### S2. Experimental

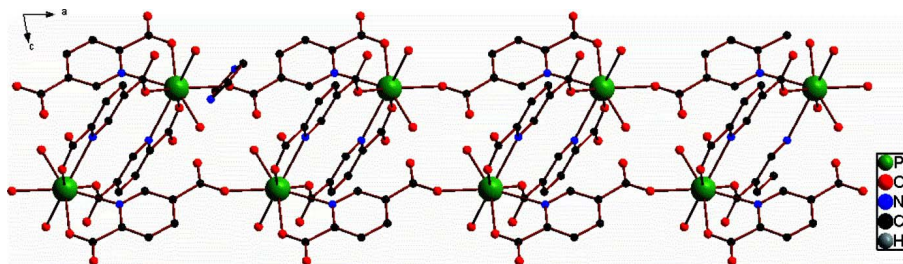
The compound **1** was synthesized by solvothermal reaction. A mixture of pyridine-2,5-dicarboxylic acid (0.0334 g, 0.2 mmol),  $\text{Pr}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$  (0.0230 g, 0.05 mmol), imidazole (0.0361 g, 0.53 mmol) and water (3 ml) was sealed in a 7 ml glass tube and heated to 120 °C for 96 h. After cooling to room temperature, light green crystals were obtained.

### S3. Refinement

Hydroxy H atoms were placed in calculated positions with  $\text{O}-\text{H} = 0.85 \text{ \AA}$ , and torsion angles were refined, Water H atoms were placed through fourier electronic density,  $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$ . Other H atoms were placed in calculated positions with  $\text{C}-\text{H} = 0.93$  (aromatic and imidazole),  $\text{N}-\text{H} = 0.86$  (imidazole) and refined in riding mode, with  $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C or N})$ .

**Figure 1**

Molecular structure of **1** showing 50% probability displacement ellipsoids.

**Figure 2**

One-dimensional chain structure of **1** viewed down the *b*-axis

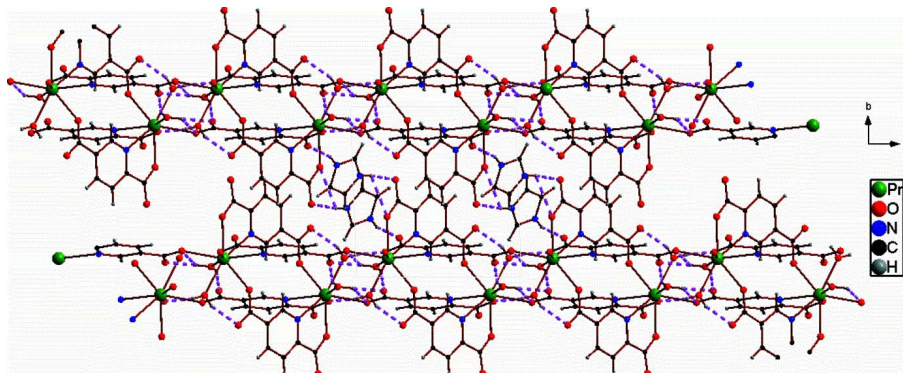


Figure 3

Hydrogen bonding packing diagram of **1** viewed down the *b*-axis.

**catena-Poly[diimidazolium [bis( $\mu$ -pyridine-2,5-dicarboxylato)bis[diaquapraseodymate(III)]]-bis( $\mu$ -pyridine-2,5-dicarboxylato)]**

#### Crystal data

$(\text{C}_3\text{H}_5\text{N}_2)_2[\text{Pr}_2(\text{C}_7\text{H}_3\text{NO}_4)_4(\text{H}_2\text{O})_4]$

$M_r = 1152.48$

Triclinic,  $P\bar{1}$

Hall symbol:  $-p\ 1$

$a = 9.5444\ (19)\ \text{\AA}$

$b = 10.667\ (2)\ \text{\AA}$

$c = 11.222\ (2)\ \text{\AA}$

$\alpha = 64.63\ (3)^\circ$

$\beta = 79.50\ (3)^\circ$

$\gamma = 87.50\ (3)^\circ$

$V = 1014.3\ (3)\ \text{\AA}^3$

$Z = 1$

$F(000) = 568$

char

$D_x = 1.887\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3561 reflections

$\theta = 2.6\text{--}25.5^\circ$

$\mu = 2.47\ \text{mm}^{-1}$

$T = 293\ \text{K}$

Block, light green

$0.19 \times 0.16 \times 0.09\ \text{mm}$

#### Data collection

Bruker SMART CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2001)

$T_{\min} = 0.652$ ,  $T_{\max} = 0.809$

14917 measured reflections

3561 independent reflections

3468 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.020$

$\theta_{\max} = 25.0^\circ$ ,  $\theta_{\min} = 2.0^\circ$

$h = -11 \rightarrow 11$

$k = -12 \rightarrow 12$

$l = -13 \rightarrow 13$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.015$

$wR(F^2) = 0.043$

$S = 1.04$

3561 reflections

305 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 atoms treated by a mixture of independent  
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0271P)^2 + 0.5143P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.068$

$\Delta\rho_{\max} = 0.33\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -0.45\ \text{e \AA}^{-3}$

#### 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 ( $\text{\AA}^2$ )*

|      | <i>x</i>      | <i>y</i>      | <i>z</i>     | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|------|---------------|---------------|--------------|----------------------------------|
| Pr1  | 0.194718 (10) | 0.107410 (10) | 0.725374 (9) | 0.01889 (5)                      |
| O1   | 0.17790 (15)  | 0.14537 (16)  | 0.92701 (14) | 0.0277 (3)                       |
| N1   | 0.43859 (18)  | 0.13489 (18)  | 0.79437 (16) | 0.0228 (4)                       |
| O4   | 0.94311 (15)  | 0.15185 (18)  | 0.73503 (16) | 0.0337 (4)                       |
| N2   | 0.36969 (19)  | 0.25158 (18)  | 0.49260 (17) | 0.0250 (4)                       |
| C5   | 0.5686 (2)    | 0.1198 (2)    | 0.7350 (2)   | 0.0252 (4)                       |
| H5   | 0.5761        | 0.0949        | 0.6642       | 0.030*                           |
| O9   | 0.0720 (2)    | −0.1109 (2)   | 0.89507 (19) | 0.0386 (4)                       |
| O5   | 0.19850 (19)  | 0.35430 (16)  | 0.63801 (16) | 0.0379 (4)                       |
| O10  | 0.1175 (2)    | 0.0591 (2)    | 0.5508 (2)   | 0.0473 (5)                       |
| C7   | 0.8356 (2)    | 0.1232 (2)    | 0.7005 (2)   | 0.0263 (5)                       |
| O3   | 0.83952 (17)  | 0.0841 (2)    | 0.60974 (18) | 0.0429 (4)                       |
| C1   | 0.4287 (2)    | 0.1698 (2)    | 0.89743 (19) | 0.0205 (4)                       |
| C6   | 0.2795 (2)    | 0.1837 (2)    | 0.9634 (2)   | 0.0248 (4)                       |
| O6   | 0.2580 (2)    | 0.57179 (18)  | 0.49073 (19) | 0.0560 (6)                       |
| C2   | 0.5464 (2)    | 0.1908 (2)    | 0.9421 (2)   | 0.0269 (5)                       |
| H2   | 0.5361        | 0.2152        | 1.0133       | 0.032*                           |
| C4   | 0.6928 (2)    | 0.1394 (2)    | 0.7731 (2)   | 0.0223 (4)                       |
| C8   | 0.3728 (2)    | 0.3897 (2)    | 0.4479 (2)   | 0.0291 (5)                       |
| O2   | 0.26769 (19)  | 0.2280 (2)    | 1.0488 (2)   | 0.0498 (5)                       |
| C13  | 0.2689 (3)    | 0.4449 (2)    | 0.5310 (2)   | 0.0325 (5)                       |
| C3   | 0.6802 (2)    | 0.1750 (2)    | 0.8796 (2)   | 0.0264 (4)                       |
| H3   | 0.7611        | 0.1882        | 0.9088       | 0.032*                           |
| C12  | 0.4576 (2)    | 0.1982 (2)    | 0.4206 (2)   | 0.0263 (4)                       |
| H12  | 0.4542        | 0.1025        | 0.4494       | 0.032*                           |
| N4   | 0.0958 (3)    | 0.3420 (3)    | 0.1892 (2)   | 0.0493 (6)                       |
| H4   | 0.1334        | 0.2930        | 0.1485       | 0.059*                           |
| C15  | −0.0147 (3)   | 0.3044 (3)    | 0.2871 (3)   | 0.0434 (6)                       |
| H15  | −0.0651       | 0.2196        | 0.3246       | 0.052*                           |
| C17  | 0.0534 (4)    | 0.5118 (3)    | 0.2475 (4)   | 0.0757 (12)                      |
| H17  | 0.0578        | 0.5961        | 0.2529       | 0.091*                           |
| N3   | −0.0426 (3)   | 0.4050 (2)    | 0.3233 (2)   | 0.0471 (6)                       |
| H3A  | −0.1110       | 0.4039        | 0.3854       | 0.056*                           |
| C16  | 0.1408 (4)    | 0.4721 (3)    | 0.1631 (4)   | 0.0760 (12)                      |
| H16  | 0.2177        | 0.5237        | 0.0987       | 0.091*                           |
| C11  | 0.5533 (2)    | 0.2772 (2)    | 0.3059 (2)   | 0.0262 (5)                       |
| C14  | 0.6545 (2)    | 0.2095 (2)    | 0.2339 (2)   | 0.0259 (4)                       |
| C10  | 0.5552 (3)    | 0.4201 (3)    | 0.2616 (3)   | 0.0439 (7)                       |
| H10  | 0.6176        | 0.4769        | 0.1845       | 0.053*                           |
| C9   | 0.4634 (3)    | 0.4765 (2)    | 0.3334 (3)   | 0.0460 (7)                       |
| H9   | 0.4624        | 0.5722        | 0.3051       | 0.055*                           |
| O8   | 0.7309 (2)    | 0.28565 (18)  | 0.12655 (17) | 0.0446 (5)                       |
| O7   | 0.65553 (16)  | 0.07807 (15)  | 0.28965 (15) | 0.0308 (3)                       |
| H9A  | 0.002 (4)     | −0.129 (3)    | 0.946 (3)    | 0.041 (8)*                       |
| H10A | 0.025 (4)     | 0.069 (3)     | 0.564 (3)    | 0.062 (10)*                      |

|      |           |            |           |             |
|------|-----------|------------|-----------|-------------|
| H10B | 0.149 (4) | 0.034 (4)  | 0.501 (4) | 0.066 (12)* |
| H9B  | 0.111 (4) | −0.174 (3) | 0.904 (3) | 0.047 (10)* |

*Atomic displacement parameters ( $\text{\AA}^2$ )*

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$    | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|-------------|--------------|
| Pr1 | 0.01230 (7) | 0.02507 (7) | 0.02203 (7) | 0.00136 (5)  | 0.00114 (5) | −0.01435 (5) |
| O1  | 0.0164 (7)  | 0.0434 (9)  | 0.0278 (7)  | −0.0005 (6)  | 0.0028 (6)  | −0.0218 (7)  |
| N1  | 0.0155 (8)  | 0.0325 (9)  | 0.0258 (8)  | 0.0028 (7)   | −0.0017 (7) | −0.0184 (7)  |
| O4  | 0.0152 (8)  | 0.0515 (10) | 0.0452 (9)  | 0.0058 (7)   | −0.0054 (7) | −0.0313 (8)  |
| N2  | 0.0237 (9)  | 0.0243 (9)  | 0.0255 (8)  | 0.0045 (7)   | 0.0023 (7)  | −0.0122 (7)  |
| C5  | 0.0183 (11) | 0.0369 (12) | 0.0272 (10) | 0.0040 (9)   | −0.0011 (8) | −0.0213 (9)  |
| O9  | 0.0263 (9)  | 0.0326 (10) | 0.0447 (10) | 0.0001 (8)   | 0.0163 (8)  | −0.0139 (8)  |
| O5  | 0.0420 (10) | 0.0259 (8)  | 0.0365 (9)  | 0.0040 (7)   | 0.0156 (7)  | −0.0139 (7)  |
| O10 | 0.0226 (10) | 0.0907 (16) | 0.0575 (12) | 0.0071 (9)   | −0.0061 (8) | −0.0598 (12) |
| C7  | 0.0170 (11) | 0.0327 (11) | 0.0320 (11) | 0.0055 (9)   | −0.0022 (9) | −0.0178 (9)  |
| O3  | 0.0201 (8)  | 0.0775 (13) | 0.0533 (10) | 0.0061 (8)   | −0.0017 (7) | −0.0512 (10) |
| C1  | 0.0184 (10) | 0.0224 (9)  | 0.0210 (9)  | 0.0024 (8)   | 0.0003 (8)  | −0.0113 (8)  |
| C6  | 0.0210 (11) | 0.0292 (11) | 0.0259 (10) | 0.0025 (9)   | 0.0015 (8)  | −0.0157 (9)  |
| O6  | 0.0734 (14) | 0.0263 (9)  | 0.0484 (11) | 0.0104 (9)   | 0.0250 (10) | −0.0122 (8)  |
| C2  | 0.0255 (11) | 0.0359 (12) | 0.0248 (10) | 0.0018 (9)   | −0.0016 (9) | −0.0192 (9)  |
| C4  | 0.0166 (10) | 0.0255 (10) | 0.0240 (10) | 0.0028 (8)   | −0.0013 (8) | −0.0111 (8)  |
| C8  | 0.0301 (12) | 0.0260 (11) | 0.0286 (11) | 0.0062 (9)   | 0.0020 (9)  | −0.0126 (9)  |
| O2  | 0.0279 (9)  | 0.0867 (14) | 0.0640 (12) | 0.0029 (9)   | 0.0032 (8)  | −0.0643 (12) |
| C13 | 0.0342 (13) | 0.0275 (12) | 0.0332 (11) | 0.0086 (10)  | 0.0041 (10) | −0.0155 (10) |
| C3  | 0.0187 (10) | 0.0351 (11) | 0.0300 (10) | 0.0024 (9)   | −0.0059 (8) | −0.0177 (9)  |
| C12 | 0.0277 (11) | 0.0218 (10) | 0.0274 (10) | 0.0045 (9)   | 0.0025 (9)  | −0.0117 (9)  |
| N4  | 0.0505 (14) | 0.0556 (14) | 0.0455 (13) | 0.0149 (12)  | 0.0073 (11) | −0.0326 (11) |
| C15 | 0.0455 (16) | 0.0406 (14) | 0.0449 (14) | 0.0077 (12)  | 0.0026 (12) | −0.0239 (12) |
| C17 | 0.080 (3)   | 0.0472 (18) | 0.089 (3)   | −0.0075 (17) | 0.039 (2)   | −0.0394 (18) |
| N3  | 0.0485 (14) | 0.0444 (12) | 0.0450 (12) | 0.0069 (11)  | 0.0155 (11) | −0.0259 (10) |
| C16 | 0.074 (2)   | 0.0543 (19) | 0.078 (2)   | −0.0050 (17) | 0.0407 (19) | −0.0288 (17) |
| C11 | 0.0247 (11) | 0.0277 (11) | 0.0249 (10) | 0.0054 (9)   | 0.0004 (9)  | −0.0122 (9)  |
| C14 | 0.0209 (11) | 0.0324 (11) | 0.0271 (10) | 0.0040 (9)   | 0.0000 (9)  | −0.0172 (9)  |
| C10 | 0.0512 (16) | 0.0282 (12) | 0.0367 (13) | 0.0020 (11)  | 0.0191 (12) | −0.0099 (10) |
| C9  | 0.0570 (18) | 0.0217 (11) | 0.0423 (14) | 0.0075 (11)  | 0.0175 (13) | −0.0089 (10) |
| O8  | 0.0481 (11) | 0.0357 (9)  | 0.0361 (9)  | 0.0047 (8)   | 0.0184 (8)  | −0.0128 (7)  |
| O7  | 0.0261 (8)  | 0.0275 (8)  | 0.0378 (8)  | 0.0042 (6)   | 0.0054 (7)  | −0.0176 (7)  |

*Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )*

|                      |             |        |           |
|----------------------|-------------|--------|-----------|
| Pr1—O5               | 2.3835 (17) | O6—C13 | 1.236 (3) |
| Pr1—O4 <sup>i</sup>  | 2.4193 (16) | C2—C3  | 1.380 (3) |
| Pr1—O7 <sup>ii</sup> | 2.4366 (16) | C2—H2  | 0.9300    |
| Pr1—O1               | 2.4407 (15) | C4—C3  | 1.385 (3) |
| Pr1—O10              | 2.4643 (18) | C8—C9  | 1.379 (3) |
| Pr1—O9               | 2.459 (2)   | C8—C13 | 1.511 (3) |
| Pr1—N1               | 2.6484 (18) | C3—H3  | 0.9300    |

|                                       |             |                      |             |
|---------------------------------------|-------------|----------------------|-------------|
| Pr1—N2                                | 2.677 (2)   | C12—C11              | 1.382 (3)   |
| O1—C6                                 | 1.263 (3)   | C12—H12              | 0.9300      |
| N1—C5                                 | 1.335 (3)   | N4—C15               | 1.310 (4)   |
| N1—C1                                 | 1.346 (3)   | N4—C16               | 1.361 (4)   |
| O4—C7                                 | 1.253 (3)   | N4—H4                | 0.8600      |
| O4—Pr1 <sup>iii</sup>                 | 2.4193 (16) | C15—N3               | 1.303 (3)   |
| N2—C8                                 | 1.337 (3)   | C15—H15              | 0.9300      |
| N2—C12                                | 1.336 (3)   | C17—C16              | 1.341 (4)   |
| C5—C4                                 | 1.384 (3)   | C17—N3               | 1.355 (4)   |
| C5—H5                                 | 0.9300      | C17—H17              | 0.9300      |
| O9—H9A                                | 0.77 (3)    | N3—H3A               | 0.8600      |
| O9—H9B                                | 0.73 (3)    | C16—H16              | 0.9300      |
| O5—C13                                | 1.263 (3)   | C11—C10              | 1.386 (3)   |
| O10—H10A                              | 0.88 (4)    | C11—C14              | 1.504 (3)   |
| O10—H10B                              | 0.73 (4)    | C14—O8               | 1.240 (3)   |
| C7—O3                                 | 1.249 (3)   | C14—O7               | 1.267 (3)   |
| C7—C4                                 | 1.499 (3)   | C10—C9               | 1.376 (3)   |
| C1—C2                                 | 1.373 (3)   | C10—H10              | 0.9300      |
| C1—C6                                 | 1.513 (3)   | C9—H9                | 0.9300      |
| C6—O2                                 | 1.224 (3)   | O7—Pr1 <sup>ii</sup> | 2.4366 (16) |
| O5—Pr1—O4 <sup>i</sup>                | 78.67 (7)   | C2—C1—C6             | 121.25 (18) |
| O5—Pr1—O7 <sup>ii</sup>               | 139.99 (6)  | O2—C6—O1             | 125.8 (2)   |
| O4 <sup>i</sup> —Pr1—O7 <sup>ii</sup> | 135.10 (6)  | O2—C6—C1             | 117.6 (2)   |
| O5—Pr1—O1                             | 77.58 (6)   | O1—C6—C1             | 116.62 (17) |
| O4 <sup>i</sup> —Pr1—O1               | 87.46 (6)   | C1—C2—C3             | 119.03 (19) |
| O7 <sup>ii</sup> —Pr1—O1              | 117.28 (6)  | C1—C2—H2             | 120.5       |
| O5—Pr1—O10                            | 104.04 (8)  | C3—C2—H2             | 120.5       |
| O4 <sup>i</sup> —Pr1—O10              | 72.69 (6)   | C3—C4—C5             | 117.72 (19) |
| O7 <sup>ii</sup> —Pr1—O10             | 75.16 (7)   | C3—C4—C7             | 121.57 (19) |
| O1—Pr1—O10                            | 159.11 (6)  | C5—C4—C7             | 120.71 (18) |
| O5—Pr1—O9                             | 145.52 (6)  | N2—C8—C9             | 122.7 (2)   |
| O4 <sup>i</sup> —Pr1—O9               | 74.82 (7)   | N2—C8—C13            | 115.37 (19) |
| O7 <sup>ii</sup> —Pr1—O9              | 73.98 (6)   | C9—C8—C13            | 121.9 (2)   |
| O1—Pr1—O9                             | 79.59 (6)   | O6—C13—O5            | 125.2 (2)   |
| O10—Pr1—O9                            | 88.76 (8)   | O6—C13—C8            | 119.1 (2)   |
| O5—Pr1—N1                             | 83.59 (7)   | O5—C13—C8            | 115.67 (19) |
| O4 <sup>i</sup> —Pr1—N1               | 148.64 (5)  | C2—C3—C4             | 119.4 (2)   |
| O7 <sup>ii</sup> —Pr1—N1              | 73.00 (6)   | C2—C3—H3             | 120.3       |
| O1—Pr1—N1                             | 63.42 (5)   | C4—C3—H3             | 120.3       |
| O10—Pr1—N1                            | 137.32 (6)  | N2—C12—C11           | 123.74 (19) |
| O9—Pr1—N1                             | 108.60 (7)  | N2—C12—H12           | 118.1       |
| O5—Pr1—N2                             | 62.40 (6)   | C11—C12—H12          | 118.1       |
| O4 <sup>i</sup> —Pr1—N2               | 117.33 (6)  | C15—N4—C16           | 108.2 (2)   |
| O7 <sup>ii</sup> —Pr1—N2              | 80.22 (6)   | C15—N4—H4            | 125.9       |
| O1—Pr1—N2                             | 124.85 (5)  | C16—N4—H4            | 125.9       |
| O10—Pr1—N2                            | 71.92 (7)   | N3—C15—N4            | 109.0 (3)   |
| O9—Pr1—N2                             | 151.07 (6)  | N3—C15—H15           | 125.5       |

|                          |             |                          |             |
|--------------------------|-------------|--------------------------|-------------|
| N1—Pr1—N2                | 75.27 (6)   | N4—C15—H15               | 125.5       |
| C6—O1—Pr1                | 125.79 (12) | C16—C17—N3               | 106.5 (3)   |
| C5—N1—C1                 | 117.88 (18) | C16—C17—H17              | 126.8       |
| C5—N1—Pr1                | 126.03 (13) | N3—C17—H17               | 126.8       |
| C1—N1—Pr1                | 116.08 (13) | C15—N3—C17               | 109.1 (2)   |
| C7—O4—Pr1 <sup>iii</sup> | 140.65 (14) | C15—N3—H3A               | 125.4       |
| C8—N2—C12                | 117.46 (18) | C17—N3—H3A               | 125.4       |
| C8—N2—Pr1                | 116.34 (13) | C17—C16—N4               | 107.2 (3)   |
| C12—N2—Pr1               | 126.14 (13) | C17—C16—H16              | 126.4       |
| N1—C5—C4                 | 123.45 (19) | N4—C16—H16               | 126.4       |
| N1—C5—H5                 | 118.3       | C12—C11—C10              | 117.9 (2)   |
| C4—C5—H5                 | 118.3       | C12—C11—C14              | 120.9 (2)   |
| Pr1—O9—H9A               | 134 (2)     | C10—C11—C14              | 121.2 (2)   |
| Pr1—O9—H9B               | 115 (3)     | O8—C14—O7                | 125.3 (2)   |
| H9A—O9—H9B               | 111 (3)     | O8—C14—C11               | 118.0 (2)   |
| C13—O5—Pr1               | 130.05 (14) | O7—C14—C11               | 116.74 (18) |
| Pr1—O10—H10A             | 103 (2)     | C9—C10—C11               | 119.0 (2)   |
| Pr1—O10—H10B             | 137 (3)     | C9—C10—H10               | 120.5       |
| H10A—O10—H10B            | 119 (4)     | C11—C10—H10              | 120.5       |
| O3—C7—O4                 | 124.8 (2)   | C10—C9—C8                | 119.3 (2)   |
| O3—C7—C4                 | 118.38 (19) | C10—C9—H9                | 120.4       |
| O4—C7—C4                 | 116.84 (19) | C8—C9—H9                 | 120.4       |
| N1—C1—C2                 | 122.48 (18) | C14—O7—Pr1 <sup>ii</sup> | 138.92 (13) |
| N1—C1—C6                 | 116.26 (18) |                          |             |

Symmetry codes: (i)  $x-1, y, z$ ; (ii)  $-x+1, -y, -z+1$ ; (iii)  $x+1, y, z$ .

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

| $D-H\cdots A$                      | $D-H$    | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|----------|-------------|-------------|---------------|
| N4—H4 $\cdots$ O2 <sup>iv</sup>    | 0.86     | 1.85        | 2.679 (3)   | 160           |
| N3—H3A $\cdots$ O6 <sup>v</sup>    | 0.86     | 1.88        | 2.735 (3)   | 172           |
| N3—H3A $\cdots$ O5 <sup>v</sup>    | 0.86     | 2.59        | 3.063 (3)   | 115           |
| O9—H9A $\cdots$ O1 <sup>vi</sup>   | 0.77 (3) | 1.98 (3)    | 2.743 (3)   | 172 (3)       |
| O10—H10A $\cdots$ O3 <sup>i</sup>  | 0.88 (4) | 1.77 (4)    | 2.645 (3)   | 173 (3)       |
| O10—H10A $\cdots$ O4 <sup>i</sup>  | 0.88 (4) | 2.44 (3)    | 2.894 (3)   | 113 (3)       |
| O10—H10B $\cdots$ O3 <sup>ii</sup> | 0.73 (4) | 2.10 (4)    | 2.789 (3)   | 157 (4)       |
| O9—H9B $\cdots$ O8 <sup>ii</sup>   | 0.73 (3) | 1.96 (4)    | 2.644 (3)   | 157 (3)       |

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