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# Synthesis and crystal structure of a new coordination polymer based on lanthanum and 1,4-phenylenediacetate ligands

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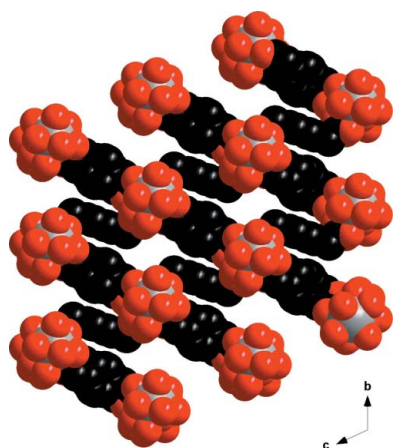
Reaction in gel between the sodium salt of 1,4-phenylenediacetic acid ( $\text{Na}_2\text{C}_{10}\text{O}_4\text{H}_8\text{-Na}_2p\text{-pda}$ ) and lanthanum chloride yields single crystals of the three-dimensional coordination polymer poly[[tetraaquatris( $\mu$ -1,4-phenylenediacetato)dilanthanum(III)] octahydrate],  $\{[\text{La}_2(\text{C}_{10}\text{H}_8\text{O}_4)_3(\text{H}_2\text{O})_4]\cdot 8\text{H}_2\text{O}\}_\infty$ . The  $\text{La}^{\text{III}}$  coordination polyhedron can be described as a slightly distorted monocapped square antiprism. One of the two  $p\text{-pda}^{2-}$  ligands is bound to four  $\text{La}^{\text{III}}$  ions and the other to two  $\text{La}^{\text{III}}$  ions. Each  $\text{La}^{\text{III}}$  atom is coordinated by five ligands, thereby generating a metal–organic framework with potential porosity properties.

## 1. Chemical context

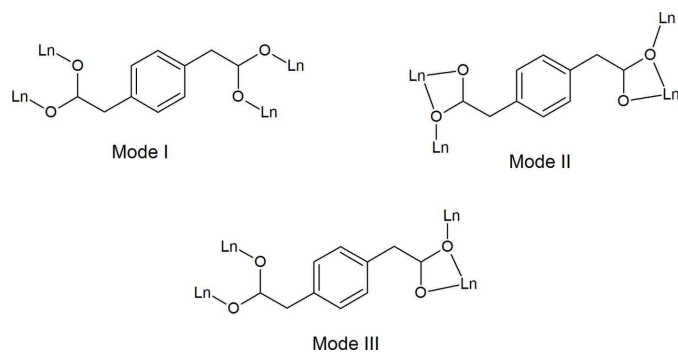
In recent years, one of the most important fields of research in coordination chemistry and crystal engineering has been the design of metal–organic frameworks (MOFs), because of their intriguing network topologies and possible applications in gas storage (Eddaoudi *et al.*, 2002; Reneike *et al.*, 1999; Luo *et al.*, 2011*a,b*; Kustaryono *et al.*, 2010), catalysis (Lee *et al.*, 2009), separation (Hamon *et al.*, 2009), luminescence (Cui *et al.*, 2012; Daiguebonne *et al.*, 2008; Binnemans, 2009;) and molecular magnetism (Calvez *et al.*, 2008; Sessoli *et al.*, 2009). Our group has been involved in this field for more than a decade (Freslon *et al.*, 2014; Fan *et al.*, 2014; Luo *et al.*, 2011*a,b*; Badiane *et al.*, 2017*a,b*). The search for new ligands that can lead to new structural networks and/or new physical properties is a continuous concern (Qiu *et al.*, 2007; Fan *et al.*, 2015).

For the synthesis of MOFs, usually two complementary molecular precursors, a cation with vacant coordination sites and a bridging anion, are used to form the coordination polymer. This procedure offers the prospect of rationally designing extended solids with interesting properties. Most of the organic ligands used in MOF chemistry are rigid aromatic carboxylates (Luo *et al.*, 2007; Huang *et al.*, 2009). Compared to the rigid ligands, using flexible ligands such as 1,2- (Xin *et al.*, 2011), 1,3- (Wang *et al.*, 2012) or 1,4-phenylenediacetate (Fabelo *et al.*, 2009*a,b*) to construct coordination polymers seems to be more difficult, and developing synthetic methodologies is still a challenge. However, flexibility of the ligand can promote structural and functional diversity.

Numerous coordination polymers have been reported so far that involve *d*-block metal ions such as  $\text{Cu}^{\text{II}}$  (Singh & Barua, 2009; Fabelo *et al.*, 2009*a,b*; Chen *et al.*, 2010*a,b,c*),  $\text{Zn}^{\text{II}}$  (Singh



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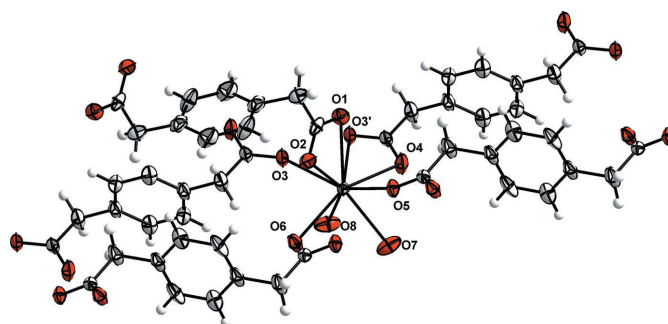
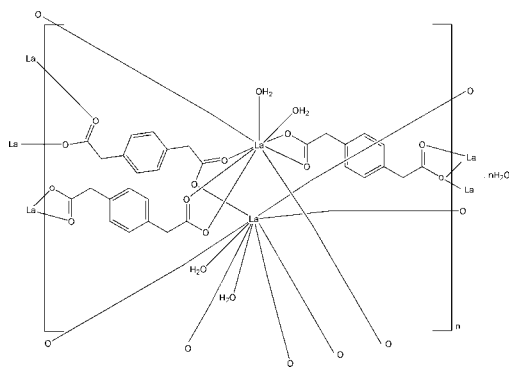


**Figure 1**  
Bonding modes in lanthanide-containing coordination polymers with 1,4-phenylenediacetate ligands ( $p\text{-pda}^{2-}$ ) reported in the literature to date.

& Barua, 2009),  $\text{Cd}^{\text{II}}$  (Chen *et al.*, 2010*a,b,c*; Singh & Barua, 2009; Li *et al.*, 2009),  $\text{Mn}^{\text{II}}$  (Singh & Barua, 2009; Chen *et al.*, 2010*a,b,c*,  $\text{Co}^{\text{II}}$  (Fabelo *et al.*, 2009*a,b*; Chen *et al.*, 2010*a,b,c*; Uebler & LaDuca, 2012; Li *et al.*, 2009) and  $\text{Ni}^{\text{II}}$  (Chen *et al.*, 2010*a,b,c*; Uebler & LaDuca, 2012; Li *et al.*, 2009). Lanthanide(III) ions have higher and variable coordination numbers (generally between 7 and 12) and incorporate in addition, apart from the main ligands, ancillary ligands such as water molecules into the lanthanide coordination sphere. A large number of studies have been reported on lanthanide coordination polymers based on 1,4-phenylenediacetic acid (Singh & Barua, 2009; Fabelo *et al.*, 2009*a,b*; Chen *et al.*, 2010*a,b,c*; Uebler & LaDuca, 2012; Li *et al.*, 2009; Rusinek *et al.*, 2013) as well as on other isomers of this acid such as 1,2- (Badiane *et al.*, 2017*a,b*; Xin *et al.*, 2011) and 1,3-phenylenediacetic acid (Wang *et al.*, 2012), and most of them tend to make porous materials through solvothermal synthesis.

Isomers of phenylenediacetic acid are flexible ligands and can therefore adopt different conformations in the crystal structure. 1,4-Phenylenediacetic acid is used as a readily available ligand that can coordinate two or more metal ions in bridging-mode, forming extended molecular networks (Pan *et al.*, 2003; Chen *et al.*, 2010*a,b,c*). The different coordination modes (Chen *et al.*, 2010*a,b,c*; Rusinek *et al.*, 2013; Ren *et al.*, 2011; Pan *et al.*, 2003; Singha *et al.*, 2014; Singha *et al.*, 2015) of the ligand with lanthanide ions that have been reported to date are shown in Fig. 1.

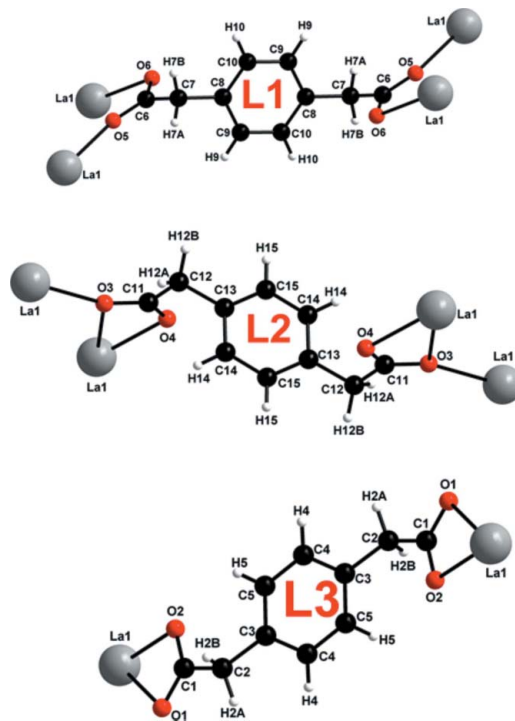
In this paper we report the synthesis and the crystal structure of a new coordination polymer with chemical formula  $[\text{La}_2(p\text{-pda})_3(\text{H}_2\text{O})_4 \cdot 8\text{H}_2\text{O}]_\infty$ .



**Figure 2**  
Coordination environment of  $\text{La}^{3+}$  in  $[\text{La}_2(p\text{-pda})_3(\text{H}_2\text{O})_4 \cdot 8\text{H}_2\text{O}]_\infty$ . Symmetry code: (')  $2 - x, 1 - y, 1 - z$ . Hydrogen atoms of the water molecules have been omitted for clarity.

## 2. Structural commentary

The crystallographically independent  $\text{La}^{3+}$  ion is nona-coordinated by seven oxygen atoms (O1, O2, O3, O4, O5, O6, O3') from five  $p\text{-pda}^{2-}$  ligands and two oxygen atoms (O8 and O7) from the coordinating water molecules (Fig. 2). The coordination polyhedron can be described as a monocapped distorted square antiprism with atom O3' capping the polyhedron [symmetry code: (')  $2 - x, 1 - y, 1 - z$ ]. The two square sides of the antiprism are formed by atoms O7, O6, O2, O5 and O8, O3, O1, O4, respectively. The dihedral angle between the two faces is  $5.21(9)^\circ$ . There are three independent ligands: **L1**, **L2** and **L3** (Fig. 3). The twisted ligand **L3** exhibits a coordination mode that has never previously been



**Figure 3**  
Coordination modes of ligand **L1** ( $\mu\text{-4}$  bis-bidentate mode:  $(\eta^1\text{-}\eta^1\text{-}\mu_2)\text{-}(\eta^1\text{-}\eta^1\text{-}\mu_2)\text{-}\mu_4$ ), **L2** ( $\mu\text{-4}$  bis-tridentate bridging and chelating mode:  $(\eta^2\text{-}\eta^1\text{-}\mu_2)\text{-}(\eta^2\text{-}\eta^1\text{-}\mu_2)\text{-}\mu_4$ ) and **L3** ( $\mu\text{-2}$  bis-bidentate-chelating mode:  $(\eta^1\text{-}\eta^1\text{-}\mu_1)\text{-}(\eta^1\text{-}\eta^1\text{-}\mu_1)\text{-}\mu_2$ ).

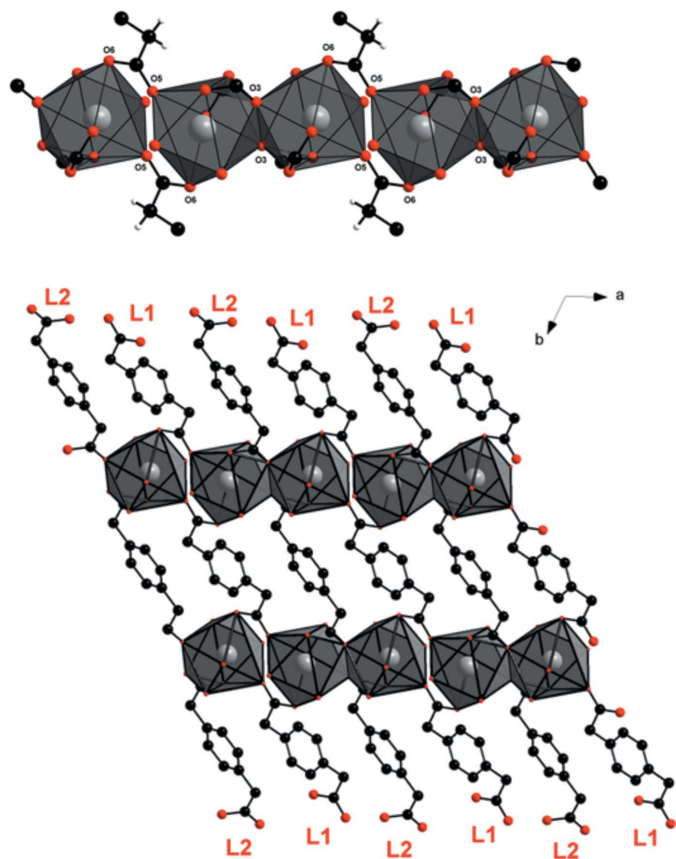
**Table 1**  
Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
OW1–HW1A···OW1 <sup>iii</sup>	0.87 (9)	2.40 (11)	3.067 (13)	133 (9)
OW1–HW1B···OW4 <sup>iv</sup>	0.89 (9)	2.54 (10)	3.298 (11)	145 (7)
OW2–HW2A···O4 <sup>i</sup>	0.82 (6)	2.10 (6)	2.895 (5)	164 (6)
OW2–HW2B···OW4 <sup>v</sup>	0.82 (6)	2.20 (5)	2.855 (8)	137 (5)
OW3–HW3A···O6 <sup>i</sup>	0.82 (8)	2.02 (8)	2.780 (8)	154 (7)
OW3–HW3B···OW1 <sup>iii</sup>	0.81 (7)	2.40 (8)	3.162 (11)	156 (8)
OW4–HW4A···OW2 <sup>vi</sup>	0.81 (10)	2.49 (9)	2.855 (8)	109 (9)
O7–H7A···O2 <sup>i</sup>	0.82 (4)	1.95 (4)	2.741 (5)	161 (5)
O7–H7B···OW4	0.81 (5)	2.03 (5)	2.800 (9)	160 (5)
OW4–HW4B···OW3 <sup>vii</sup>	0.84 (9)	2.11 (10)	2.824 (11)	143 (8)
O8–H8A···OW3 <sup>i</sup>	0.82 (4)	2.38 (4)	3.175 (8)	165 (4)
O8–H8B···O1 <sup>ii</sup>	0.83 (4)	1.92 (4)	2.725 (5)	163 (5)
C7–H7D···O4 <sup>i</sup>	0.97	2.54	3.442 (6)	154
C12–H12B···O6 <sup>ii</sup>	0.97	2.51	3.406 (6)	154

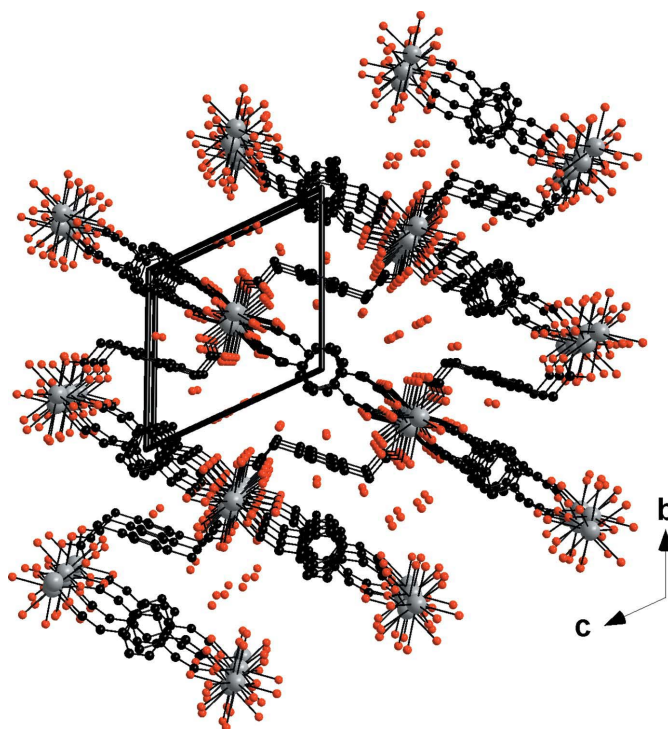
Symmetry codes: (i)  $-x+1, -y+1, -z+1$ ; (ii)  $-x+2, -y+1, -z+1$ ; (iii)  $-x+1, -y, -z+1$ ; (iv)  $x, y-1, z$ ; (v)  $x, y, z+1$ ; (vi)  $x, y, z-1$ ; (vii)  $x+1, y+1, z$ .

observed in lanthanide-based coordination polymers involving the *p*-pda<sup>2-</sup> ligand.

The monocapped square antiprisms are connected to each other by alternating **L1** bridging carboxylate oxygen atoms (O5 and O6) and edge-sharing polyhedra through **L2** oxygen atoms (O3), forming molecular chains along the *a*-axis direction (Fig. 4). These chains are connected to each other through

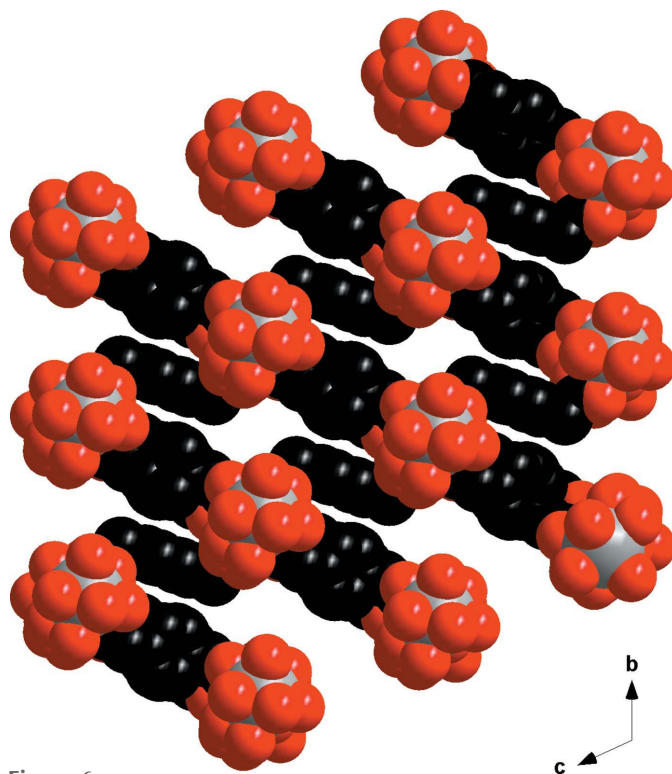


**Figure 4**  
(Top) Projection view of a molecular chain extending parallel to the *a* axis. (Bottom) Projection view along the *c* axis of the of the two-dimensional molecular network of  $[\text{La}_2(\text{p-pda})_3(\text{H}_2\text{O})_4 \cdot 8\text{H}_2\text{O}]_\infty$ . Hydrogen atoms have been omitted for clarity.



**Figure 5**  
Perspective view along the *a* axis of  $[\text{La}_2(\text{p-pda})_3(\text{H}_2\text{O})_4 \cdot 8\text{H}_2\text{O}]_\infty$ . Hydrogen atoms have been omitted for clarity.

ligands **L1** and **L2**, which play the role of spacers, forming molecular layers that extend parallel to the *ab* plane (Fig. 4).



**Figure 6**  
Projection view along the *a* axis of the molecular skeleton of  $[\text{La}_2(\text{p-pda})_3(\text{H}_2\text{O})_4 \cdot 8\text{H}_2\text{O}]_\infty$  in space-filling mode. Hydrogen atoms and crystallization water molecules have been omitted.

These layers are further connected through the twisted ligand **L3**, leading to a three-dimensional molecular framework (Fig. 5). Ligand **L3** acts as a spacer between the different polymeric layers because of its *anti-anti* conformation.

The framework has channels along the *a*-axis direction in which the water molecules of crystallization are located. They are bound to the molecular skeleton *via* a hydrogen-bonded network (Table 1). As can be seen from Fig. 6, the three-dimensional crystal structure could potentially present some porosity properties. Indeed, removal of the water molecules of crystallization could create empty channels, as has been reported previously (Kustaryono *et al.*, 2010; Kerbellec *et al.*, 2008). For the coordination polymer in this study, the potential porosity is calculated to be  $750(20) \text{ m}^2 \text{ g}^{-1}$  for  $\text{N}_2$  with a kinetic radius of 1.83 Å. The calculation was performed using a method described elsewhere (Kustaryono *et al.*, 2010; Kerbellec *et al.*, 2008).

Other crystal structures of lanthanide coordination polymers with the *p*-pda<sup>2-</sup> ligand have been reported previously. This series of compounds, first described by Pan *et al.* (2003) has been widely studied because of potential applications in various fields such as explosives detection (Singha *et al.*, 2014, 2015), gas sorption (Pan *et al.*, 2003) or catalysis (Ren *et al.*, 2011). These compounds, with general chemical formula  $[\text{Ln}_2(\text{p-pda})_3(\text{H}_2\text{O})\cdot 2\text{H}_2\text{O}]_\infty$  with  $\text{Ln} = \text{La-Ho}$  have been obtained by hydrothermal synthesis and therefore present a lower hydration rate and a higher density than  $[\text{La}_2(\text{p-pda})_3(\text{H}_2\text{O})_4\cdot 8\text{H}_2\text{O}]_\infty$   $\{D_{\text{calc}} = 1871 \text{ g cm}^{-3}$  for  $[\text{Ln}_2(\text{p-pda})_3(\text{H}_2\text{O})\cdot 2\text{H}_2\text{O}]_\infty\}$ . Their three-dimensional crystal structures can be described on the basis of helicoidal molecular chains linked by *p*-pda<sup>2-</sup> ligands.

The luminescent and porosity properties of these compounds are interesting, which suggests that the physical properties of compounds isostructural to  $[\text{La}_2(\text{p-pda})_3(\text{H}_2\text{O})_4\cdot 8\text{H}_2\text{O}]_\infty$  and involving other lanthanide ions (lanthanum is a diamagnetic non-luminescent ion) would be worth studying. Unfortunately, despite great synthetic efforts, no such compound has been obtained to date.

The compound reported here was obtained by crystallization in a gel (see next section; Luo *et al.*, 2013), and as such is the first result from our group related to lanthanide-based coordination polymers with 1,4-phenylenediacetate ligands.

### 3. Synthesis and crystallization

Lanthanum oxide ( $\text{La}_2\text{O}_3$ ) was suspended in a small quantity of water. The suspension was then brought to about 323 K and concentrated hydrochloric acid was added dropwise under magnetic stirring, until a clear solution was obtained. The solution was then evaporated to dryness and the resulting solid was dissolved in absolute ethanol for removal of the residual hydrochloric acid. Crystallization of the salt was then obtained by adding diethyl ether ( $\text{Et}_2\text{O}$ ). The obtained microcrystalline solid was filtered and dried in the open air. The product  $\text{LaCl}_3\cdot 7\text{H}_2\text{O}$  was obtained in close to 100% yield.

1,4-Phenylenediacetic acid,  $\text{H}_2(\text{p-pda})$ , was purchased from Sigma-Aldrich and used without further purification. Its

**Table 2**  
Experimental details.

Crystal data	
Chemical formula	$[\text{La}_2(\text{C}_{10}\text{H}_8\text{O}_4)_3(\text{H}_2\text{O})_4]\cdot 8\text{H}_2\text{O}$
$M_r$	1070.05
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	293
$a, b, c$ (Å)	9.1197 (2), 11.1231 (2), 11.9434 (2)
$\alpha, \beta, \gamma$ (°)	107.049 (1), 107.729 (1), 106.622 (1)
$V$ (Å <sup>3</sup> )	1005.21 (3)
$Z$	1
Radiation type	Mo $K\alpha$
$\mu$ (mm <sup>-1</sup> )	2.18
Crystal size (mm)	0.08 × 0.06 × 0.05
Data collection	
Diffractometer	Nonius KappaCCD
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	4588, 4588, 3751
$R_{\text{int}}$	0.045
$(\sin \theta/\lambda)_{\text{max}}$ (Å <sup>-1</sup> )	0.649
Refinement	
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.030, 0.068, 1.03
No. of reflections	4588
No. of parameters	283
No. of restraints	18
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$ (e Å <sup>-3</sup> )	1.76, -0.65

Computer programs: *COLLECT* (Bruker, 2004), *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997), *SHELXT* (Sheldrick, 2015a), *SHELXL* (Sheldrick, 2015b), *DIAMOND* (Brandenburg, 2001), *WinGX* (Farrugia, 2012).

disodium salt was prepared by addition of two equivalents of sodium hydroxide to a suspension of the acid in de-ionized water. The obtained clear solution was evaporated to dryness and then refluxed in ethanol for one h. Addition of diethyl ether provoked precipitation of  $\text{Na}_2(\text{p-pda})$  in 90% yield. UV-vis absorption spectrum of a  $4.3 \times 10^{-4} \text{ mol L}^{-1}$  aqueous solution of the disodium salt of  $\text{H}_2(\text{p-pda})$  was measured with a Perkin-Elmer Lambda 650 spectrometer equipped with a 60 mm integrating sphere. It showed a maximum absorption at 225 nm. This short absorption wavelength, compared to other ligands in the literature (Badiane *et al.*, 2017a,b; Freslon *et al.*, 2016; Fan *et al.*, 2015; Badiane *et al.*, 2018), can be related to the  $-\text{CH}_2-$  groups that cut conjugation.

Single crystals of the coordination polymer were obtained by slow diffusion of dilute aqueous solutions of lanthanum chloride (0.25 mmol in 10 mL) and of the sodium salt of *para*-phenylenediacetate (0.25 mmol in 10 mL) through an agar-agar gel in a U-shaped tube. The gel was purchased from Acros Organics and jellified according to established procedures (Henisch, 1988; Daguebonne *et al.*, 2003). After several weeks, prismatic single crystals were obtained.

### 4. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. Hydrogen atoms bound to the organic ligands were placed at idealized positions ( $\text{C-H} = 0.93\text{--}0.97 \text{ Å}$ ) and refined as riding with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

The water hydrogen atoms were localized and constrained. The thermal agitation of the two water molecules of crystallization was constrained. In order to stabilize the refinement several restraints (DANG, DFIX) were used for the hydrogen atoms bound to water oxygens.

### Acknowledgements

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## supporting information

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## Synthesis and crystal structure of a new coordination polymer based on lanthanum and 1,4-phenylenediacetate ligands

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### Computing details

Data collection: *COLLECT* (Bruker, 2004); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *DIAMOND* (Brandenburg, 2001); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

### Poly[[tetraaquatris( $\mu$ -1,4-phenylenediacetato)dilanthanum(III)] octahydrate]

#### Crystal data

$[\text{La}_2(\text{C}_{10}\text{H}_8\text{O}_4)_3(\text{H}_2\text{O})_4] \cdot 8\text{H}_2\text{O}$

$M_r = 1070.05$

Triclinic,  $P\bar{1}$

Hall symbol: -P 1

$a = 9.1197$  (2) Å

$b = 11.1231$  (2) Å

$c = 11.9434$  (2) Å

$\alpha = 107.049$  (1)°

$\beta = 107.729$  (1)°

$\gamma = 106.622$  (1)°

$V = 1005.21$  (3) Å<sup>3</sup>

$Z = 1$

$F(000) = 534$

$D_x = 1.768$  Mg m<sup>-3</sup>

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

Cell parameters from 15558 reflections

$\theta = 2.9$ – $27.5$ °

$\mu = 2.18$  mm<sup>-1</sup>

$T = 293$  K

Prism, colorless

$0.08 \times 0.06 \times 0.05$  mm

#### Data collection

Nonius KappaCCD  
diffractometer

Radiation source: Enraf Nonius FR590

Horizontally mounted graphite crystal  
monochromator

Detector resolution: 9 pixels mm<sup>-1</sup>

CCD rotation images, thick slices scans

4588 measured reflections

4588 independent reflections

3751 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.045$

$\theta_{\text{max}} = 27.5$ °,  $\theta_{\text{min}} = 3.6$ °

$h = -10 \rightarrow 11$

$k = -14 \rightarrow 14$

$l = -15 \rightarrow 15$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.068$

$S = 1.03$

4588 reflections

283 parameters

18 restraints

0 constraints

Primary atom site location: dual

Secondary atom site location: dual

Hydrogen site location: mixed

H atoms treated by a mixture of independent  
and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0308P)^2]$

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

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

$$\Delta\rho_{\max} = 1.76 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.65 \text{ e } \text{\AA}^{-3}$$

### Special details

**Geometry.** All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
La1	0.75090 (2)	0.52031 (2)	0.47969 (2)	0.02048 (7)
O5	0.4472 (3)	0.3677 (2)	0.4069 (2)	0.0332 (6)
O3	1.0575 (3)	0.5881 (3)	0.6253 (2)	0.0323 (6)
O1	0.7627 (3)	0.3041 (3)	0.5311 (2)	0.0336 (6)
O6	0.8146 (3)	0.7579 (3)	0.6435 (3)	0.0362 (6)
O2	0.7422 (3)	0.4674 (3)	0.6736 (2)	0.0351 (6)
O4	0.6840 (3)	0.3616 (3)	0.2490 (2)	0.0415 (7)
O7	0.5690 (4)	0.5882 (4)	0.3233 (3)	0.0486 (8)
H7A	0.468 (3)	0.558 (4)	0.307 (5)	0.058*
H7B	0.591 (5)	0.651 (4)	0.302 (5)	0.058*
O8	0.9316 (4)	0.7051 (3)	0.4326 (4)	0.0503 (8)
H8A	0.947 (6)	0.785 (3)	0.470 (4)	0.060*
H8B	1.026 (4)	0.712 (4)	0.435 (5)	0.060*
C6	0.6834 (4)	0.7426 (3)	0.6621 (3)	0.0260 (7)
C11	0.8165 (4)	0.3436 (3)	0.2659 (3)	0.0261 (7)
C1	0.7640 (4)	0.3589 (4)	0.6405 (4)	0.0315 (8)
C13	0.6579 (5)	0.1147 (4)	0.0739 (4)	0.0340 (9)
C12	0.8247 (5)	0.2382 (4)	0.1565 (4)	0.0390 (9)
H12A	0.857342	0.281262	0.102898	0.058*
H12B	0.910563	0.207990	0.192279	0.058*
C14	0.6017 (5)	0.0190 (4)	0.1196 (4)	0.0427 (10)
H14	0.669560	0.030195	0.201149	0.051*
C8	0.8472 (5)	0.9325 (4)	0.8881 (4)	0.0340 (9)
C10	0.9564 (5)	1.0646 (4)	0.9217 (4)	0.0433 (10)
H10	0.928413	1.109955	0.869797	0.052*
C15	0.5547 (5)	0.0946 (4)	-0.0469 (4)	0.0412 (10)
H15	0.589046	0.157191	-0.080797	0.049*
C7	0.6832 (5)	0.8578 (4)	0.7675 (4)	0.0445 (11)
H7C	0.660724	0.923722	0.734480	0.067*
H7D	0.592152	0.819798	0.789973	0.067*
C9	0.8927 (6)	0.8687 (4)	0.9685 (4)	0.0461 (11)
H9	0.820475	0.779508	0.948162	0.055*
C3	0.8997 (5)	0.3972 (4)	0.8732 (4)	0.0392 (9)
C4	1.0738 (6)	0.4427 (5)	0.9267 (4)	0.0514 (11)
H4	1.125405	0.405378	0.878263	0.062*
C5	0.8281 (6)	0.4561 (5)	0.9477 (4)	0.0501 (11)
H5	0.711419	0.427472	0.913133	0.060*



C2	0.7931 (6)	0.2928 (4)	0.7347 (4)	0.0460 (10)
H2A	0.684726	0.235879	0.727251	0.069*
H2B	0.848359	0.233001	0.711571	0.069*
OW1	0.6313 (10)	0.0072 (5)	0.4440 (6)	0.1327 (17)
HW1A	0.589 (11)	-0.025 (9)	0.491 (8)	0.159*
HW1B	0.639 (13)	-0.072 (6)	0.411 (8)	0.159*
OW2	0.5351 (5)	0.6185 (4)	0.9718 (4)	0.0741 (11)
HW2A	0.476 (7)	0.639 (6)	0.920 (5)	0.111*
HW2B	0.622 (6)	0.678 (5)	1.033 (5)	0.111*
OW3	0.0357 (9)	0.0056 (6)	0.3870 (6)	0.1327 (17)
HW3A	0.059 (10)	0.059 (7)	0.354 (7)	0.159*
HW3B	0.122 (7)	0.023 (10)	0.447 (6)	0.159*
OW4	0.7105 (10)	0.7873 (7)	0.2433 (6)	0.154 (3)
HW4A	0.649 (10)	0.805 (11)	0.192 (8)	0.185*
HW4B	0.810 (5)	0.831 (11)	0.255 (10)	0.185*

*Atomic displacement parameters (Å<sup>2</sup>)*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
La1	0.01729 (10)	0.02001 (10)	0.01850 (10)	0.00633 (7)	0.00482 (7)	0.00498 (7)
O5	0.0242 (13)	0.0258 (13)	0.0312 (13)	0.0032 (10)	0.0034 (11)	0.0040 (11)
O3	0.0227 (13)	0.0340 (14)	0.0229 (12)	0.0074 (11)	0.0027 (10)	0.0011 (11)
O1	0.0352 (14)	0.0353 (14)	0.0339 (14)	0.0161 (12)	0.0168 (12)	0.0155 (12)
O6	0.0247 (13)	0.0283 (13)	0.0415 (15)	0.0089 (10)	0.0129 (12)	-0.0005 (11)
O2	0.0403 (15)	0.0427 (15)	0.0332 (14)	0.0254 (13)	0.0173 (12)	0.0200 (12)
O4	0.0267 (14)	0.0515 (17)	0.0253 (13)	0.0175 (12)	0.0040 (11)	-0.0052 (12)
O7	0.0425 (17)	0.075 (2)	0.067 (2)	0.0390 (17)	0.0345 (17)	0.0532 (18)
O8	0.0456 (18)	0.0523 (18)	0.088 (2)	0.0315 (16)	0.0434 (18)	0.0469 (19)
C6	0.0228 (17)	0.0219 (17)	0.0260 (17)	0.0100 (14)	0.0051 (14)	0.0052 (14)
C11	0.0187 (16)	0.0250 (17)	0.0236 (16)	0.0060 (13)	0.0062 (14)	0.0018 (14)
C1	0.0184 (17)	0.041 (2)	0.0332 (19)	0.0099 (15)	0.0083 (15)	0.0177 (17)
C13	0.0261 (19)	0.030 (2)	0.0297 (19)	0.0117 (16)	0.0070 (16)	-0.0041 (16)
C12	0.0233 (18)	0.040 (2)	0.0318 (19)	0.0098 (16)	0.0074 (16)	-0.0057 (17)
C14	0.039 (2)	0.044 (2)	0.028 (2)	0.0198 (19)	0.0002 (18)	0.0047 (18)
C8	0.031 (2)	0.0293 (19)	0.0290 (19)	0.0102 (16)	0.0138 (16)	-0.0032 (16)
C10	0.042 (2)	0.033 (2)	0.037 (2)	0.0058 (18)	0.0114 (19)	0.0059 (18)
C15	0.043 (2)	0.037 (2)	0.031 (2)	0.0133 (19)	0.0076 (18)	0.0078 (17)
C7	0.026 (2)	0.040 (2)	0.043 (2)	0.0127 (17)	0.0102 (18)	-0.0078 (19)
C9	0.042 (2)	0.0225 (19)	0.048 (2)	-0.0033 (17)	0.016 (2)	0.0006 (18)
C3	0.043 (2)	0.044 (2)	0.035 (2)	0.0206 (19)	0.0123 (18)	0.0260 (18)
C4	0.051 (3)	0.073 (3)	0.041 (2)	0.037 (2)	0.022 (2)	0.023 (2)
C5	0.032 (2)	0.077 (3)	0.042 (2)	0.024 (2)	0.0112 (19)	0.027 (2)
C2	0.055 (3)	0.040 (2)	0.039 (2)	0.017 (2)	0.012 (2)	0.0233 (19)
OW1	0.192 (4)	0.069 (2)	0.109 (3)	0.009 (3)	0.079 (3)	0.028 (2)
OW2	0.063 (3)	0.086 (3)	0.074 (3)	0.032 (2)	0.020 (2)	0.043 (2)
OW3	0.192 (4)	0.069 (2)	0.109 (3)	0.009 (3)	0.079 (3)	0.028 (2)
OW4	0.158 (6)	0.115 (4)	0.103 (4)	-0.017 (4)	-0.019 (4)	0.084 (4)

*Geometric parameters* (Å, °)

La1—O5	2.507 (2)	C12—H12B	0.9700
La1—O5 <sup>i</sup>	2.905 (3)	C14—H14	0.9300
La1—O3 <sup>ii</sup>	2.781 (2)	C14—C15 <sup>iii</sup>	1.399 (6)
La1—O3	2.545 (2)	C8—C10	1.373 (6)
La1—O1	2.673 (2)	C8—C7	1.509 (5)
La1—O6	2.559 (2)	C8—C9	1.388 (6)
La1—O2	2.569 (2)	C10—H10	0.9300
La1—O4	2.566 (2)	C10—C9 <sup>iv</sup>	1.382 (6)
La1—O7	2.543 (3)	C15—H15	0.9300
La1—O8	2.562 (3)	C7—H7C	0.9700
La1—C11	3.066 (3)	C7—H7D	0.9700
La1—C1	2.988 (4)	C9—H9	0.9300
O5—C6 <sup>i</sup>	1.255 (4)	C3—C4	1.385 (6)
O3—C11 <sup>ii</sup>	1.264 (4)	C3—C5	1.381 (6)
O1—C1	1.264 (4)	C3—C2	1.509 (6)
O6—C6	1.256 (4)	C4—H4	0.9300
O2—C1	1.248 (4)	C4—C5 <sup>v</sup>	1.391 (6)
O4—C11	1.246 (4)	C5—H5	0.9300
O7—H7A	0.824 (19)	C2—H2A	0.9700
O7—H7B	0.806 (18)	C2—H2B	0.9700
O8—H8A	0.815 (19)	OW1—HW1A	0.87 (2)
O8—H8B	0.833 (19)	OW1—HW1B	0.88 (2)
C6—C7	1.512 (5)	OW2—HW2A	0.821 (19)
C11—C12	1.516 (5)	OW2—HW2B	0.81 (2)
C1—C2	1.514 (5)	OW3—HW3A	0.82 (2)
C13—C12	1.507 (5)	OW3—HW3B	0.81 (2)
C13—C14	1.376 (6)	OW4—HW4A	0.81 (2)
C13—C15	1.371 (5)	OW4—HW4B	0.84 (2)
C12—H12A	0.9700		
O5—La1—O5 <sup>i</sup>	61.48 (9)	C11—O4—La1	101.3 (2)
O5—La1—O3	146.45 (9)	La1—O7—H7A	116 (3)
O5—La1—O3 <sup>ii</sup>	118.58 (7)	La1—O7—H7B	133 (3)
O5—La1—O1	75.87 (8)	H7A—O7—H7B	109 (3)
O5—La1—O6	107.86 (8)	La1—O8—H8A	118 (3)
O5—La1—O2	75.29 (8)	La1—O8—H8B	122 (3)
O5—La1—O4	80.33 (8)	H8A—O8—H8B	104 (3)
O5—La1—O7	71.93 (9)	O5 <sup>i</sup> —C6—La1	68.28 (18)
O5—La1—O8	140.13 (9)	O5 <sup>i</sup> —C6—O6	120.2 (3)
O5 <sup>i</sup> —La1—C11	156.28 (8)	O5 <sup>i</sup> —C6—C7	120.1 (3)
O5—La1—C11	98.48 (8)	O6—C6—La1	52.33 (16)
O5—La1—C1	75.84 (9)	O6—C6—C7	119.7 (3)
O5 <sup>i</sup> —La1—C1	88.69 (9)	C7—C6—La1	169.6 (2)
O3—La1—O5 <sup>i</sup>	118.22 (7)	O3 <sup>ii</sup> —C11—La1	65.08 (17)
O3 <sup>ii</sup> —La1—O5 <sup>i</sup>	179.00 (7)	O3 <sup>ii</sup> —C11—C12	120.3 (3)
O3—La1—O3 <sup>ii</sup>	61.11 (8)	O4—C11—La1	55.16 (17)

O3—La1—O1	73.20 (8)	O4—C11—O3 <sup>ii</sup>	119.9 (3)
O3—La1—O6	80.93 (8)	O4—C11—C12	119.8 (3)
O3—La1—O2	74.87 (8)	C12—C11—La1	171.2 (2)
O3—La1—O4	108.82 (8)	O1—C1—La1	63.39 (19)
O3—La1—O8	73.39 (9)	O1—C1—C2	119.8 (3)
O3 <sup>ii</sup> —La1—C11	24.36 (8)	O2—C1—La1	58.58 (18)
O3—La1—C11	85.47 (8)	O2—C1—O1	121.5 (3)
O3—La1—C1	70.64 (9)	O2—C1—C2	118.7 (3)
O3 <sup>ii</sup> —La1—C1	90.37 (9)	C2—C1—La1	172.4 (3)
O1—La1—O5 <sup>i</sup>	109.68 (7)	C14—C13—C12	120.5 (4)
O1—La1—O3 <sup>ii</sup>	69.49 (8)	C15—C13—C12	122.1 (4)
O1—La1—C11	74.29 (9)	C15—C13—C14	117.4 (4)
O1—La1—C1	25.01 (9)	C11—C12—H12A	109.2
O6—La1—O5 <sup>i</sup>	46.41 (7)	C11—C12—H12B	109.2
O6—La1—O3 <sup>ii</sup>	133.56 (8)	C13—C12—C11	112.0 (3)
O6—La1—O1	126.21 (9)	C13—C12—H12A	109.2
O6—La1—O2	78.86 (9)	C13—C12—H12B	109.2
O6—La1—O4	149.49 (10)	H12A—C12—H12B	107.9
O6—La1—O8	71.55 (10)	C13—C14—H14	119.1
O6—La1—C11	149.62 (9)	C13—C14—C15 <sup>iii</sup>	121.8 (4)
O6—La1—C1	101.90 (10)	C15 <sup>iii</sup> —C14—H14	119.1
O2—La1—O5 <sup>i</sup>	66.57 (8)	C10—C8—C7	121.6 (4)
O2—La1—O3 <sup>ii</sup>	112.44 (8)	C10—C8—C9	117.8 (4)
O2—La1—O1	49.39 (8)	C9—C8—C7	120.6 (4)
O2—La1—C11	123.42 (9)	C8—C10—H10	119.5
O2—La1—C1	24.50 (9)	C8—C10—C9 <sup>iv</sup>	121.1 (4)
O4—La1—O5 <sup>i</sup>	132.95 (7)	C9 <sup>iv</sup> —C10—H10	119.5
O4—La1—O3 <sup>ii</sup>	47.75 (7)	C13—C15—C14 <sup>iii</sup>	120.7 (4)
O4—La1—O1	84.13 (9)	C13—C15—H15	119.6
O4—La1—O2	131.27 (9)	C14 <sup>iii</sup> —C15—H15	119.6
O4—La1—C11	23.49 (8)	C6—C7—H7C	108.9
O4—La1—C1	108.61 (10)	C6—C7—H7D	108.9
O7—La1—O5 <sup>i</sup>	70.81 (9)	C8—C7—C6	113.4 (3)
O7—La1—O3	141.53 (10)	C8—C7—H7C	108.9
O7—La1—O3 <sup>ii</sup>	110.19 (9)	C8—C7—H7D	108.9
O7—La1—O1	142.43 (10)	H7C—C7—H7D	107.7
O7—La1—O6	82.59 (10)	C8—C9—H9	119.4
O7—La1—O2	134.87 (9)	C10 <sup>iv</sup> —C9—C8	121.1 (4)
O7—La1—O4	71.95 (10)	C10 <sup>iv</sup> —C9—H9	119.4
O7—La1—O8	68.46 (10)	C4—C3—C2	120.7 (4)
O7—La1—C11	91.69 (10)	C5—C3—C4	118.0 (4)
O7—La1—C1	147.21 (10)	C5—C3—C2	121.2 (4)
O8—La1—O5 <sup>i</sup>	108.04 (8)	C3—C4—H4	119.8
O8—La1—O3 <sup>ii</sup>	72.59 (9)	C3—C4—C5 <sup>v</sup>	120.4 (4)
O8—La1—O1	138.11 (8)	C5 <sup>v</sup> —C4—H4	119.8
O8—La1—O2	139.28 (10)	C3—C5—C4 <sup>v</sup>	121.6 (4)
O8—La1—O4	83.41 (11)	C3—C5—H5	119.2
O8—La1—C11	78.57 (10)	C4 <sup>v</sup> —C5—H5	119.2

O8—La1—C1	144.03 (10)	C1—C2—H2A	109.0
C1—La1—C11	98.95 (10)	C1—C2—H2B	109.0
La1—O5—La1 <sup>i</sup>	118.52 (9)	C3—C2—C1	112.8 (3)
C6 <sup>i</sup> —O5—La1 <sup>i</sup>	88.1 (2)	C3—C2—H2A	109.0
C6 <sup>i</sup> —O5—La1	153.4 (2)	C3—C2—H2B	109.0
La1—O3—La1 <sup>ii</sup>	118.89 (8)	H2A—C2—H2B	107.8
C11 <sup>ii</sup> —O3—La1	150.5 (2)	HW1A—OW1—HW1B	88 (7)
C11 <sup>ii</sup> —O3—La1 <sup>iii</sup>	90.56 (19)	HW2A—OW2—HW2B	121 (5)
C1—O1—La1	91.6 (2)	HW3A—OW3—HW3B	108 (4)
C6—O6—La1	104.8 (2)	HW4A—OW4—HW4B	107 (4)
C1—O2—La1	96.9 (2)		

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
OW1—HW1A $\cdots$ OW1 <sup>vi</sup>	0.87 (9)	2.40 (11)	3.067 (13)	133 (9)
OW1—HW1B $\cdots$ OW4 <sup>vii</sup>	0.89 (9)	2.54 (10)	3.298 (11)	145 (7)
OW2—HW2A $\cdots$ O4 <sup>i</sup>	0.82 (6)	2.10 (6)	2.895 (5)	164 (6)
OW2—HW2B $\cdots$ OW4 <sup>viii</sup>	0.82 (6)	2.20 (5)	2.855 (8)	137 (5)
OW3—HW3A $\cdots$ O6 <sup>i</sup>	0.82 (8)	2.02 (8)	2.780 (8)	154 (7)
OW3—HW3B $\cdots$ OW1 <sup>vi</sup>	0.81 (7)	2.40 (8)	3.162 (11)	156 (8)
OW4—HW4A $\cdots$ OW2 <sup>ix</sup>	0.81 (10)	2.49 (9)	2.855 (8)	109 (9)
O7—H7A $\cdots$ O2 <sup>i</sup>	0.82 (4)	1.95 (4)	2.741 (5)	161 (5)
O7—H7B $\cdots$ OW4	0.81 (5)	2.03 (5)	2.800 (9)	160 (5)
OW4—HW4B $\cdots$ OW3 <sup>x</sup>	0.84 (9)	2.11 (10)	2.824 (11)	143 (8)
O8—H8A $\cdots$ OW3 <sup>i</sup>	0.82 (4)	2.38 (4)	3.175 (8)	165 (4)
O8—H8B $\cdots$ O1 <sup>ii</sup>	0.83 (4)	1.92 (4)	2.725 (5)	163 (5)
C7—H7D $\cdots$ O4 <sup>i</sup>	0.97	2.54	3.442 (6)	154
C12—H12B $\cdots$ O6 <sup>ii</sup>	0.97	2.51	3.406 (6)	154

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