

Shi-Jie Huang, Dan-Yi Wei and Yue-Qing Zheng*

State Key Laboratory Base of Novel Functional Materials and Preparation Science, Institute of Solid Materials Chemistry, Faculty of Materials Science and Chemical Engineering, Ningbo University, Ningbo 315211, People's Republic of China

Correspondence e-mail:
 zhengyueqing@nbu.edu.cn

Key indicators

Single-crystal X-ray study
 T = 298 K
 Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
 R factor = 0.019
 wR factor = 0.053
 Data-to-parameter ratio = 18.1

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

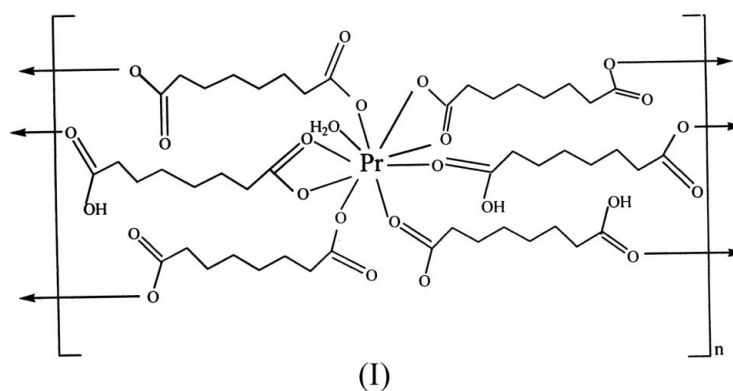
Poly[[aquapraseodymium(III)]- μ -(8-carboxy-octanoato)- μ -octanedioato]

Received 12 August 2006
 Accepted 1 September 2006

The asymmetric unit in the title complex, $[\text{Pr}(\text{C}_8\text{H}_{12}\text{O}_4)(\text{C}_8\text{H}_{13}\text{O}_4)(\text{H}_2\text{O})]_n$, comprises a Pr^{3+} cation, a suberate dianion (L^{2-}), a protonated suberate anion (HL^-) and one aqua ligand. The L^{2-} and HL^- ligands show similar coordination modes. The crystal structure features infinite praseodymium–oxygen chains of edge-shared (PrO_9) polyhedra connected by carbon backbones to form a three-dimensional framework.

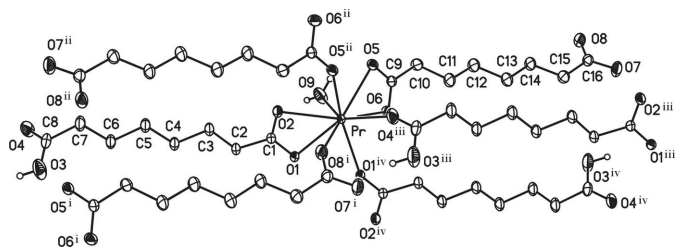
Comment

Due to their conformational and coordination versatility, aliphatic α,ω -dicarboxylate ligands have been recognized as important flexible spacers in the construction of supramolecular polymers (Dimos *et al.*, 2002; Serpaggi & Férey 2003; Rao *et al.*, 2004; Zheng & Xie, 2004). The title complex, (I), was synthesized under hydrothermal conditions and is isostructural with $[\text{La}(\text{C}_8\text{H}_{13}\text{O}_4)(\text{C}_8\text{H}_{12}\text{O}_4)(\text{OH}_2)]_n$ (Benmerad *et al.*, 2004).

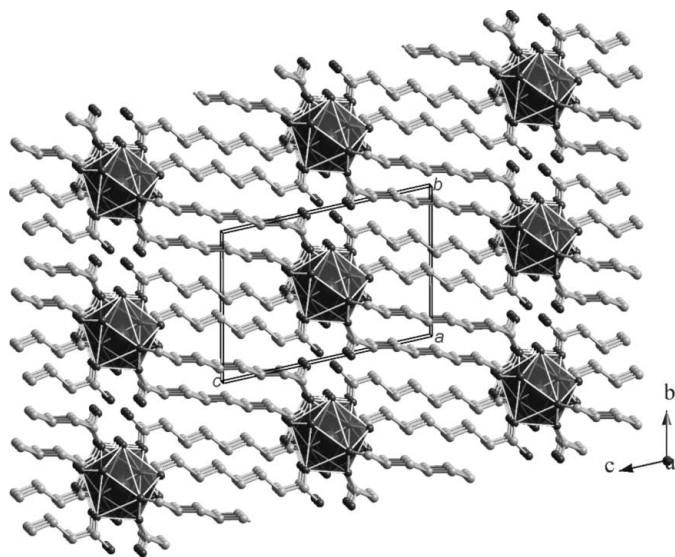


The crystallographic asymmetric unit of (I) (Fig. 1) comprises a Pr^{3+} cation, a suberate dianion (L^{2-}), a protonated suberate anion (HL^-) and one aqua ligand. The Pr^{3+} cation is sited within a distorted monocapped dodecahedron defined by nine O atoms derived from six different bridging ligands and a coordinated water molecule. Two of the carboxylate groups, *viz.* one derived from L^{2-} and the other from HL^- , are chelating, and the remaining four carboxylates coordinate in a monodentate mode. The $\text{Pr}-\text{O}$ bond distances lie in the range 2.438 (2)–2.644 (2) Å (Table 1). The bond lengths and angles within the suberate anions exhibit normal values (Zheng & Kong, 2002; Zheng & Xie, 2004; Wei *et al.*, 2002; Thalladi *et al.*, 2000).

The PrO_9 coordination polyhedra are edge-shared through two carboxylate O atoms (O1 of HL^- and O5 of L^{2-}) to generate infinite praseodymium–oxygen chains extending in the [100] direction (Fig. 2). Within these chains, the distances

**Figure 1**

The coordination geometry about the Pr^{3+} cation in (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 45% probability level. All but oxygen-bound H atoms have been omitted for clarity. Symmetry operators are as given in Table 1.

**Figure 2**

A projection of the packing of (I), along the [100] direction. All H atoms have been omitted for clarity.

between neighbouring Pr^{3+} ions are almost equal [4.275 (1) (connection across atom O5) and 4.229 (2) Å (connection across atom O1)], which shows that there are no interactions between neighbouring Pr^{3+} cations. The resulting chains are further connected by the HL^{2-} and L^- ligands to form a three-dimensional framework, which can be described as inorganic layers of praseodymium oxide chains pillared by organic spacers (Fig. 2).

The crystal structure of (I) is stabilized by hydrogen-bonding interactions between the coordinated water and carboxylate O atoms, of both L and HL ligands, as detailed in Table 2.

Experimental

Pr_2O_3 (0.049 g, 0.15 mmol) was dissolved in concentrated HCl (4.0 ml) and the solution heated nearly to dryness. An aqueous solution (10 ml) of suberic acid (0.052 g, 0.30 mmol) was then added. The mixture was then adjusted to pH 4.00 using NaOH (1.0 M) and the resulting mixture was transferred to a 23 ml Teflon-lined stainless steel autoclave, which was heated at 443 K for 72 h. Green crystals of (I) were grown after slow cooling.

Crystal data

$[\text{Pr}(\text{C}_8\text{H}_{12}\text{O}_4)(\text{C}_8\text{H}_{13}\text{O}_4)(\text{H}_2\text{O})]$
 $M_r = 504.29$
 Triclinic, $P\bar{1}$
 $a = 8.4690$ (17) Å
 $b = 9.0086$ (18) Å
 $c = 13.100$ (3) Å
 $\alpha = 100.55$ (3)°
 $\beta = 103.95$ (3)°
 $\gamma = 97.85$ (3)°

$V = 936.3$ (4) Å³
 $Z = 2$
 $D_x = 1.789$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 2.65$ mm⁻¹
 $T = 298$ (2) K
 Block, green
 0.28 × 0.22 × 0.21 mm

Data collection

Rigaku R-Axis RAPID
 diffractometer
 ω scans
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.502$, $T_{\max} = 0.574$

9320 measured reflections
 4249 independent reflections
 4072 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.012$
 $\theta_{\max} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.019$
 $wR(F^2) = 0.053$
 $S = 1.16$
 4249 reflections
 235 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0218P)^2 + 1.4812P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.10$ e Å⁻³
 $\Delta\rho_{\min} = -0.46$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

| | | | |
|---------------------------------------|------------|--|------------|
| Pr—O1 | 2.600 (2) | Pr—O5 ⁱⁱⁱ | 2.440 (2) |
| Pr—O1 ⁱ | 2.466 (2) | Pr—O6 | 2.530 (2) |
| Pr—O2 | 2.555 (2) | Pr—O8 ^{iv} | 2.438 (2) |
| Pr—O4 ⁱⁱ | 2.451 (2) | Pr—O9 | 2.503 (2) |
| Pr—O5 | 2.644 (2) | | |
| O1—Pr—O1 ⁱ | 66.82 (7) | O2—Pr—O6 | 140.60 (8) |
| O1—Pr—O2 | 50.38 (7) | O2—Pr—O8 ^{iv} | 73.70 (8) |
| O1—Pr—O4 ⁱⁱ | 145.28 (8) | O2—Pr—O9 | 69.77 (8) |
| O1—Pr—O5 | 138.15 (7) | O4 ⁱⁱ —Pr—O5 | 74.11 (8) |
| O1—Pr—O5 ⁱⁱⁱ | 124.79 (6) | O4 ⁱⁱ —Pr—O5 ⁱⁱⁱ | 74.63 (8) |
| O1—Pr—O6 | 114.20 (7) | O4 ⁱⁱ —Pr—O6 | 74.11 (9) |
| O1—Pr—O8 ^{iv} | 74.90 (8) | O4 ⁱⁱ —Pr—O8 ^{iv} | 80.01 (8) |
| O1—Pr—O9 | 71.26 (8) | O4 ⁱⁱ —Pr—O9 | 138.78 (8) |
| O1 ⁱ —Pr—O2 | 115.40 (7) | O5—Pr—O5 ⁱⁱⁱ | 65.57 (7) |
| O1 ⁱ —Pr—O4 ⁱⁱ | 84.72 (8) | O5—Pr—O6 | 49.77 (7) |
| O1 ⁱ —Pr—O5 | 123.85 (6) | O5—Pr—O8 ^{iv} | 143.73 (7) |
| O1 ⁱ —Pr—O5 ⁱⁱⁱ | 154.05 (7) | O5—Pr—O9 | 66.91 (8) |
| O1 ⁱ —Pr—O6 | 74.68 (7) | O5 ⁱⁱⁱ —Pr—O6 | 113.30 (7) |
| O1 ⁱ —Pr—O8 ^{iv} | 77.52 (7) | O5 ⁱⁱⁱ —Pr—O8 ^{iv} | 83.47 (8) |
| O1 ⁱ —Pr—O9 | 105.87 (9) | O5 ⁱⁱⁱ —Pr—O9 | 100.04 (9) |
| O2—Pr—O4 ⁱⁱ | 141.63 (9) | O6—Pr—O8 ^{iv} | 143.34 (8) |
| O2—Pr—O5 | 113.09 (7) | O6—Pr—O9 | 70.85 (9) |
| O2—Pr—O5 ⁱⁱⁱ | 75.04 (7) | O8 ^{iv} —Pr—O9 | 140.85 (8) |

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

Table 2

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|-------|-------------|-------------|---------------|
| O3—H3 \cdots O7 ^v | 0.85 | 1.67 | 2.441 (4) | 149 |
| O9—H9A \cdots O7 ^{vi} | 0.82 | 2.11 | 2.816 (4) | 144 |
| O9—H9B \cdots O3 ^{vii} | 0.82 | 2.47 | 2.987 (4) | 122 |

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

Carbon-bound H atoms were included in the riding-model approximation, with C–H = 0.97 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. Oxygen-bound H atoms were initially located in a difference map but were then fixed in the riding-model approximation, with O–H = 0.82–0.85 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$ (hydroxyl-O) or $1.5U_{\text{eq}}$ (water-O). The highest residual electron-density peak of $1.10 \text{ e } \text{Å}^{-3}$ was located 0.80 Å from the Pr atom.

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP II* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Expert Project of Key Basic Research of the Ministry of Science and Technology of China (grant No. 2003CCA00800), the Zhejiang Provincial Natural Science Foundation (grant No. Z203067) and Ningbo Municipal Science Foundation (grant No. 2006 A610061)

References

- Benmerad, B., Guehria-Laidoudi, A., Dahaoui, S. & Lecomte, C. (2004). *Acta Cryst.* **C60**, m407–m409.
- Dimos, A., Tsaousis, D., Michaelides, A., Skoulika, S., Golhen, S., Ouahab, L., Didierjean, C. & Aubry, A. (2002). *Chem. Mater.* **14**, 2616–2622.
- Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
- Johnson, C. K. (1976). *ORTEP II*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Rao, C. N. R., Natarajan, S. & Vaidyanathan, R. (2004). *Angew. Chem. Int. Ed.* **43**, 1466–1496.
- Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
- Rigaku/MS (2002). *CrystalStructure*. Rigaku/MS Inc., The Woodlands, Texas, USA.
- Serpaggi, F. & Férey, G. (2003). *J. Mol. Struct.* **656**, 201–206.
- Sheldrick, G. M. (1990). *SHELXS97*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Thalladi, V. R., Nüsse, M. & Boese, R. (2000). *J. Am. Chem. Soc.* **122**, 9227–9236.
- Wei, D. Y., Zheng, Y. Q. & Lin, J. L. (2002). *Z. Anorg. Allg. Chem.* **628**, 2005–2012.
- Zheng, Y. Q. & Kong, Z. P. (2002). *J. Solid State Chem.* **166**, 279–284.
- Zheng, Y. Q. & Xie, H. Z. (2004). *J. Solid State Chem.* **177**, 1352–1358.