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Poly[[diaqua[μ_6-(butane-1,4-diyldinitrilotetramethylene)tetraphosphonato]di-μ_2-oxalato-digadolinium(III)] tetrahydrate]

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Key indicators

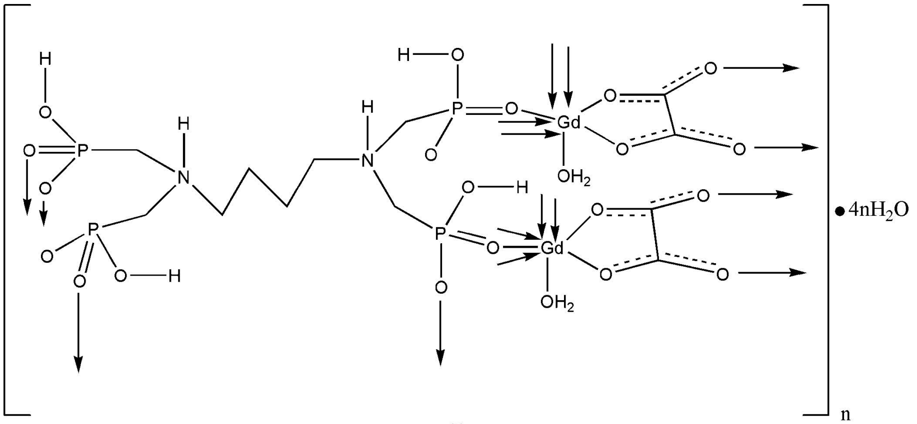
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.013 Å
R factor = 0.045
wR factor = 0.101
Data-to-parameter ratio = 12.4

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

The title compound, $[Gd_2(C_8H_{22}N_2O_{12}P_4)(C_2O_4)_2(H_2O)_2]\cdot 4H_2O$, was synthesized by the hydrothermal method. It exhibits a pillar–layer structure, with the layers formed by Gd^{III} ions interconnected by the oxalate ions and the $-NH(CH_2PO_3H)_2$ units of the tetraphosphonate dianions. The central organic groups of the tetraphosphonate ligands act as the pillars. Each oxalate ion bridges two Gd^{III} ions and each tetraphosphonate ligand bridges six Gd^{III} ions. Inversion centres are located at the mid-points of the central C—C bonds of both types of ligands.

Comment

Inorganic–organic hybrid materials based on metal phosphonates are interesting because of their potential applications in electro-optics, ion exchange and catalysis, and as sensors (Clearfield, 1998). Metal phosphonates have been reported (Clearfield, 1998; Cheetham *et al.*, 1999; Stock *et al.*, 2000; Distler & Sevov, 1998; Ying *et al.*, 2006), but studies on lanthanide phosphonates are scarce and are mainly based on X-ray powder diffraction (Serpaggi & Ferey, 1999; Ngo & Lin, 2002; Nash *et al.*, 1998). Lanthanide phosphonates have low solubilities in water and other organic solvents, making it difficult to obtain single crystals suitable for X-ray structural analysis. The introduction of a second ligand such as 5-sulfoisophthalic or oxalic acid improves the solubility and crystallinity of such compounds (Song *et al.*, 2004; Ying & Mao, 2006).



(I)

In the title compound, (I) (Fig. 1), the Gd^{III} ion is eightfold coordinated by four O atoms from two oxalate anions, three phosphonate O atoms from three tetraphosphonate anions, and an aqua ligand. The coordination geometry of the metal ion can be described as a distorted bicapped trigonal prism.

The complexes assemble in a pillar–layer structure, with the layers formed by Gd^{III} ions interconnected by the oxalate ions and the $–NH(CH_2PO_3H)_2$ units of the tetraphosphonate dianions. The central organic groups of the tetraphosphonate ligand act as the pillars (Fig. 2). Inversion centres are located at the mid-points of the central C—C bonds of both ligands.

This compound is isostructural with the analogous Nd^{III} and Eu^{III} compounds reported previously by Ying & Mao (2006). The structure of (I) is further reinforced through an extensive three-dimensional hydrogen-bond network (Table 1).

Experimental

A mixture of Gd_2O_3 (0.2 mmol, 0.070 g), H_8L [H_8L = $(H_2O_3PCH_2)_2N(CH_2)_4N(CH_2PO_3H_2)_2$] (0.2 mmol, 0.090 g), and $H_2C_2O_4$ (0.40 mmol, 0.051 g) in distilled water (10 ml) was sealed in an autoclave equipped with a Teflon liner (20 ml) and then heated at 453 K for 5 d. Crystals of the title compound were obtained as colourless needles.

Crystal data

$[Gd_2(C_8H_{22}N_2O_{12}P_4)(C_2O_4)_2(H_2O)_2]\cdot 4H_2O$
M_r = 530.40
Triclinic, $P\overline{1}$
a = 9.5191 (6) Å
b = 9.6282 (6) Å
c = 9.6452 (6) Å
α = 106.939 (3)°
β = 98.292 (3)°
γ = 110.123 (2)°
V = 764.05 (8) $Å^3$
Z = 2
D_x = 2.305 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 4.62 mm^{-1}
T = 293 (2) K
Needle, colourless
0.25 × 0.04 × 0.03 mm

Data collection

Bruker SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.387, T_{max} = 0.893
6629 measured reflections
2728 independent reflections
2140 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 25.2°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.101
S = 0.94
2728 reflections
220 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0597P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.78 e $Å^{-3}$
$\Delta\rho_{min}$ = −1.92 e $Å^{-3}$
Extinction correction: *SHELXL* (Sheldrick, 1997)
Extinction coefficient: 0.0049 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O11^{i}	0.82	1.81	2.628 (8)	177
O13—H13B···O11^{ii}	0.82	1.94	2.751 (10)	168
O4—H4···O12^{iii}	0.82	1.81	2.602 (8)	161
O11—H11B···O10^{iv}	0.86	1.87	2.720 (8)	172
O12—H12A···O6^{ii}	0.85	1.92	2.752 (10)	165
O11—H11A···O6^{v}	0.84	1.99	2.805 (9)	161

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

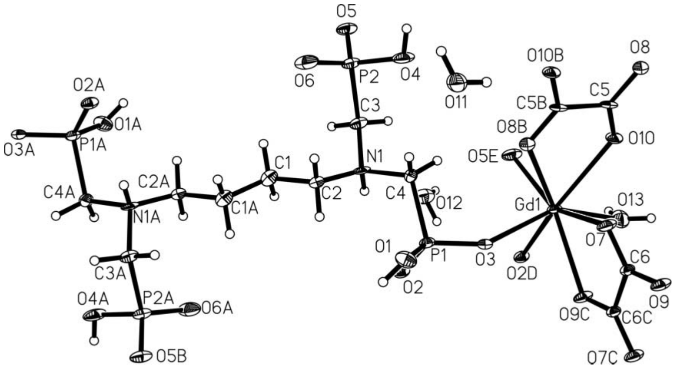


Figure 1
Part of the structure of the title compound, with 30% probability displacement ellipsoids. [Symmetry codes: (A) $-x, -y+1, -z$; (B) $-x+1, -y+2, -z+2$; (C) $-x+1, -y+1, -z+2$; (D) $-x+1, -y+1, -z+1$; (E) $-x+1, -y+2, -z+1$.]

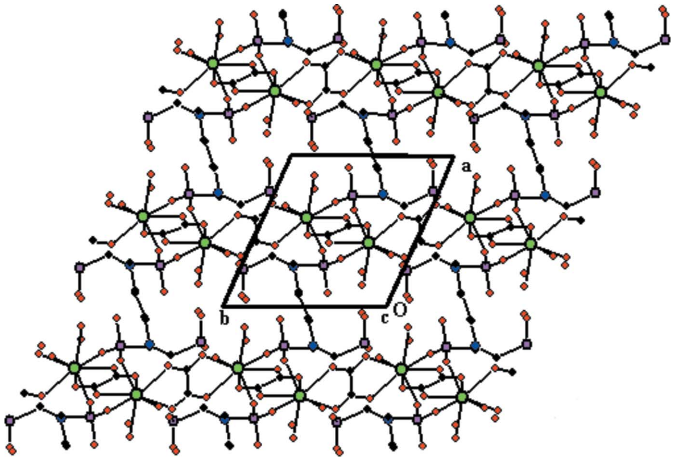


Figure 2
A projection of the title compound along the c axis. Gd, P, N, C and O atoms are represented by green, purple, blue, black and red circles, respectively. H atoms have been omitted for clarity.

All C-bound H atoms were generated geometrically, with C—H distances ranging from 0.93 to 0.98 Å, and refined in a riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}$ of the carrier atom. The remaining H atoms were found in a difference Fourier map and refined isotropically, with $U_{iso}(H) = 1.2U_{eq}$ of the carrier atom. The maximum residual electron density was observed 0.96 Å from atom Gd1 and the minimum residual electron density was observed 0.90 Å from the same atom. The residual electron-density features and some highly anisotropic displacement ellipsoids are probably a consequence of an imperfect absorption correction.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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References

Bruker (2004). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Cheetham, A. K., Ferey, G. & Loiseau, T. (1999). *Angew. Chem. Int. Ed.* **38**, 3268–3292.

Clearfield, A. (1998). *Metal Phosphonate Chemistry*. In *Progress in Inorganic Chemistry*, edited by K. D. Karlin, Vol. 47, pp. 371–510, and references therein. New York: John Wiley & Sons.

Distler, A. & Sevov, S. C. (1998). *Chem. Commun.* pp. 959–960.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Nash, K. L., Rogers, R. D., Ferraro, J. & Zhang, J. (1998). *Inorg. Chim. Acta*, **269**, 211–213.

Ngo, H. L. & Lin, W.-B. (2002). *J. Am. Chem. Soc.* **124**, 14298–14299.

Serpaggi, F. & Ferey, G. (1999). *Inorg. Chem.* **38**, 4741–4744.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Sheldrick, G. M. (2003). *SADABS*. Version 2.10. Bruker AXS Inc., Madison, Wisconsin, USA.

Song, J.-L., Lei, C. & Mao, J.-G. (2004). *Inorg. Chem.* **43**, 5630–5634.

Stock, N., Frey, S. A., Stucky, G. D. & Cheetham, A. K. (2000). *J. Chem. Soc. Dalton Trans.* pp. 4292–4294.

Ying, S.-M. & Mao, J.-G. (2006). *Cryst. Growth Des.* **6**, 964–968.

Ying, S.-M., Zeng, X.-R., Fang, X.-N., Li, X.-F. & Liu, D.-S. (2006). *Inorg. Chim. Acta*, **359**, 1589–1593.

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