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Poly[[bis[pentaaquagadolinium(III)]-μ_4-benzene-1,2,3,4,5,6-hexacarboxylato] tetrahydrate]

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The title compound, $\{[Gd_2(C_{12}O_{12})(H_2O)_{10}]\cdot 4H_2O\}_n$, consists of an extended network of Gd ions coordinated by the mellitate anions and water molecules. In this complex, each Gd atom involves a dodecahedral coordination environment comprising five water molecules and three O atoms from two separate mellitate anions. The center of mass of the hexaanion $[C_6(COO)_6]^{6-}$ coincides with a crystallographic inversion center.

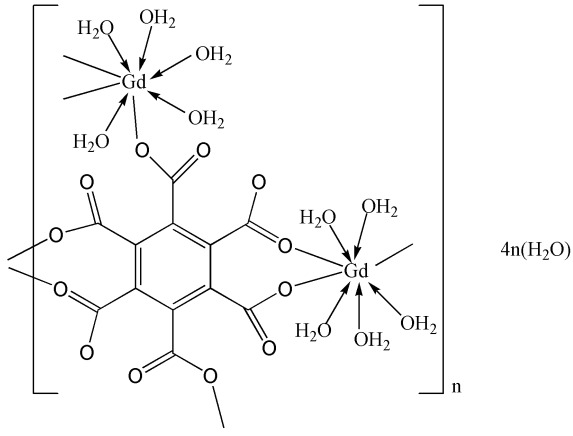
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.007 Å
R factor = 0.029
wR factor = 0.083
Data-to-parameter ratio = 14.8

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

Comment

The title compound, (I), is isostructural with its $[M_2\{C_6(COO)_6\}(H_2O)_{10}]\cdot 4H_2O$ [M = Y; Robl & Hentschel, 1992), Yb (Wu *et al.*, 1996) and Er (Deluzet & Guillou, 2003) analogs. As illustrated in Fig. 1, the Gd atom possesses dodecahedral coordination geometry (Table 1), in which the Gd—O bond distances range from 2.327 (3) to 2.425 (3) Å, with an average bond distance of 2.393 Å, similar to those found in the previously reported isostructural complexes.



(I)

A crystallographic inversion center is located at the center of the benzene ring of the mellitate ligand. Each of the six carboxylate groups displays a monodentate coordination mode and those attached to atoms C3 and C5 bind the same Gd^{III} ion, forming a seven-membered chelate ring. In this mode, each mellitate group bridges four metal centers, yielding a two-dimensional sheet structure. The carboxylate C atoms deviate from the least-squares plane of the benzene ring by 0.012 (8)–0.090 (8) Å. The dihedral angle between the mean plane of the monodentate carboxylate group and the

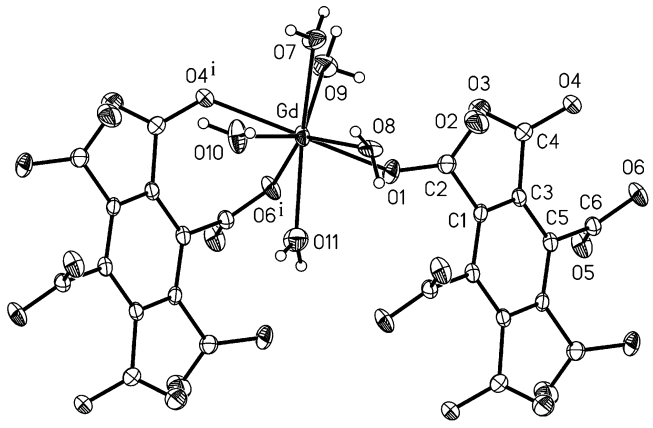


Figure 1
The coordination environment of the Gd^{III} ion in (I), showing 45% probability displacement ellipsoids. [Symmetry code: (i) $x-1, y, z$.]

benzene ring ring varies little, ranging from 51.8 (5) to 71.5 (4)°. Such an unstrained conformation of the mellitate anion is presumably an essential element in respect of the arrangement of the two-dimensional sheet structure.

All of the water molecules are involved in a complex hydrogen-bonding network. Each of the water molecules has at least one hydrogen-bonding interaction (Table 2), and these make a significant contribution to the stability of the crystal structure.

Experimental

For the synthesis, $GdCl_3{\cdot}6H_2O$ (0.38 g, 1.0 mmol) was added to a stirred solution of mellitic acid (0.11 g, 0.5 mmol) and NaOH (0.03 g, 1.5 mmol) in CH_3OH/H_2O (50 ml, 1:1 *v*/*v*). The resulting mixture was further stirred for *ca* 30 min to yield a colorless solution, which was then maintained at 323 K, and colorless crystals grew after two days.

Crystal data

$[Gd_2(C_{12}O_{12})(H_2O)_{10}]{\cdot}4H_2O$
$M_r = 451.42$
Monoclinic, $P2_1/n$
$a = 8.510$ (1) Å
$b = 9.277$ (1) Å
$c = 16.464$ (2) Å
$\beta = 100.337$ (2)°
$V = 1278.7$ (3) Å^3
$Z = 4$
$D_x = 2.345$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 156 reflections
$\theta = 1.8$–26.5°
$\mu = 5.26$ mm^{-1}
$T = 295$ (2) K
Column, colorless
0.21 × 0.12 × 0.08 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.479$, $T_{max} = 0.663$
6721 measured reflections
2701 independent reflections
2448 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 26.8°$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 12$
$l = -8 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.083$
$S = 1.02$
2701 reflections
182 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0525P)^2 + 5.4257P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.88$ e Å^{-3}
$\Delta\rho_{min} = -0.64$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0016 (4)

Table 1
Selected bond lengths (Å).

Gd—O1	2.327 (3)	Gd—O8	2.421 (5)
Gd—O4^{i}	2.402 (3)	Gd—O9	2.402 (4)
Gd—O6^{i}	2.398 (3)	Gd—O10	2.425 (3)
Gd—O7	2.380 (5)	Gd—O11	2.391 (4)

Symmetry code: (i) $x-1, y, z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
O7—H7A···O13^{ii}	0.82	2.05	2.818 (8)	155
O8—H8B···O3^{iii}	0.82	2.06	2.835 (4)	158
O9—H9A···O2^{ii}	0.82	2.21	2.911 (3)	143
O10—H10A···O2^{i}	0.82	2.10	2.897 (3)	163
O10—H10B···O4^{iii}	0.82	1.97	2.783 (5)	174
O11—H11A···O13^{i}	0.82	1.96	2.762 (7)	167
O11—H11B···O1^{iv}	0.82	2.45	3.199 (5)	152
O12—H12A···O5^{v}	0.82	1.92	2.74 (1)	175
O13—H13A···O3^{v}	0.82	2.10	2.919 (4)	176
O13—H13B···O12^{vi}	0.82	2.38	3.08 (2)	143

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+\frac{1}{2}, y+\frac{1}{2}, -z+\frac{3}{2}$; (iii) $-x+\frac{1}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$; (iv) $-x, -y+1, -z+1$; (v) $-x+1, -y+1, -z+1$; (vi) $x+1, y, z$.

All H atoms were located in difference maps and then treated as riding atoms, with O—H distances of 0.82 Å and with $U_{iso}(H) = 0.05$ Å^2.

Data collection: *SMART* (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: *SHELXTL* (Bruker, 2004); software used to prepare material for publication: *SHELXTL*.

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References

Bruker (2004). *SMART* (Version 6.0), *SAINT* (Version 6.0) and *SHELXTL* (Version 5.1). Bruker AXS Inc., Madison, Wisconsin, USA.
Deluzet, A. & Guillou, O. (2003). *Acta Cryst.* C**59**, m277–m279.
Robl, C. & Hentschel, S. (1992). *Z. Naturforsch. Teil B*, **47**, 1561–1565.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Wu, L. P., Munakata, M., Kuroda-Sowa, T., Maekawa, M. & Suenage, Y. (1996). *Inorg. Chim. Acta*, **249**, 183–189.