catena-Poly[[bis[pentaaqua-erbium(III)]-µ-benzenehexa-carboxylato] tetrahydrate]

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The title compound is composed of one-dimensional polymeric $\{[Er_2(C_{12}O_{12})(H_2O)_{10}]\cdot 4H_2O\}_n$ chains containing Er in a slightly distorted antiprismatic eightfold coordination. The benzenehexacarboxylate ion is located about an inversion centre. Water molecules of crystallization, linked by hydrogen bonding to water molecules of the rare earth coordination spheres or the carboxylate groups of the organic ligands, fill the space generated by the packing of the separated chains.

Comment

Aromatic acids usually form rare earth salts which exhibit optical properties (strong fluorescent emission) and possess very important physical and biological properties. Mellitic acid (benzenehexacarboxylic acid) can lose between two to six H atoms, depending on the reaction conditions used, and this gives rise to a great diversity of salts. The mellitate anion is known to be a multidentate agent via its carboxylate O atoms to metal cations, such as main group metal ions (for example, Ca or Al; Uchtman & Jandacek, 1980; Giacovazzo et al., 1973), transition metal ions (Cu[II], Ni[II], Co[III] or Y[III]; Endres & Knieszner, 1984; Robl & Hentschel, 1991, 1992) or rare earth ions (La[III]; Wu et al., 1996). In these materials, the dimensionality of the networks varies from three-dimensional in $[Al_2\{C_6(CO_2)_6\}]\cdot 16H_2O$ to two-dimensional in the Cu compound, where layers are linked to each other by hydrogen bonds, and one-dimensional in the Co or Ni compounds, where infinite chains are formed. We present here the crystal structure of the title compound, (I), an analogous Er compound with mellitate.

The title compound, (I), forms a one-dimensional polymer. As shown in Fig. 1, the chains are formed by the succession of one organic ligand and a pair of Er atoms along **a**. Each chain is separated from neighbouring chains by free water molecules (Fig. 2).

The Er atom in (I) is eight-coordinated by three O atoms from two mellitate ions and five water O atoms. The Er—O distances are quite homogeneous (Table 1) and the environment of the Er atom is a slightly distorted square antiprism (Fig. 3). The mellitate ion is located about an inversion centre

(I)
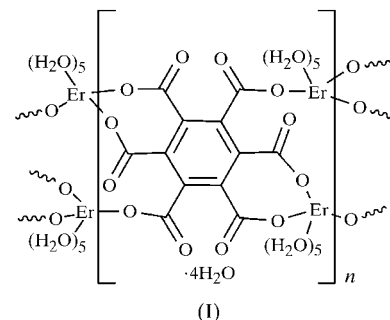
and each ligand is bonded to four different Er atoms. The six carboxylate groups are each linked to an Er atom via one O atom, while the other O atom remains free. The carboxylate groups are greatly twisted out of the plane of the benzene ring
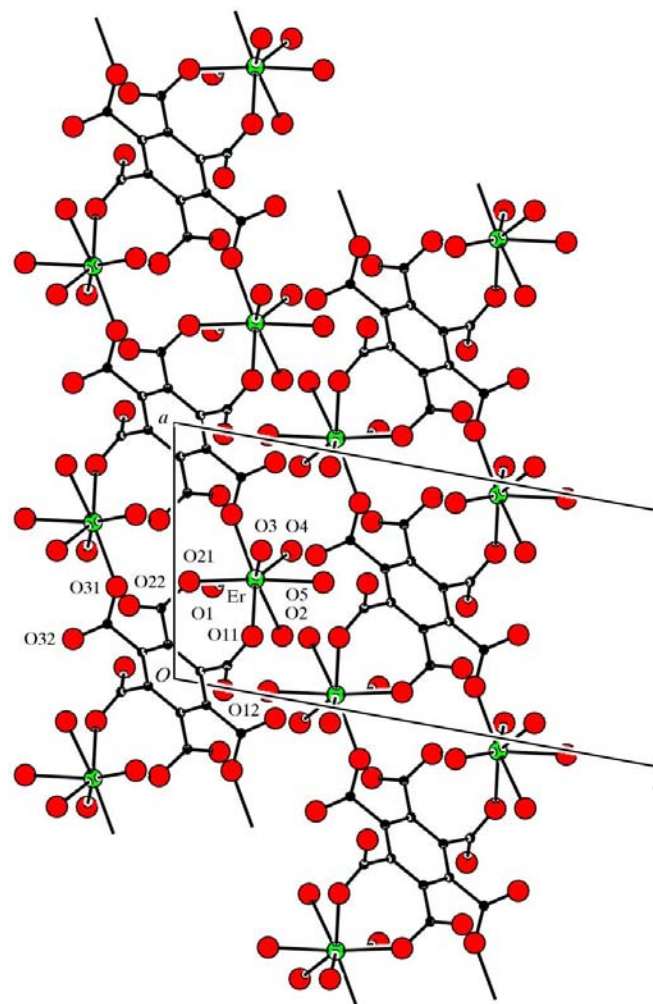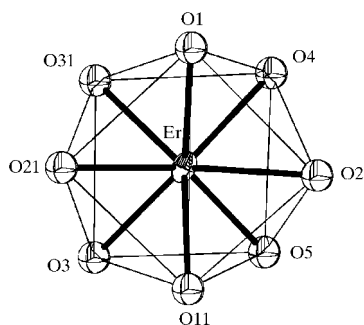

Figure 1
A projection view of (I) along the b axis, showing the packing of the infinite chains. For clarity, water molecules of crystallization and all H atoms have been omitted.
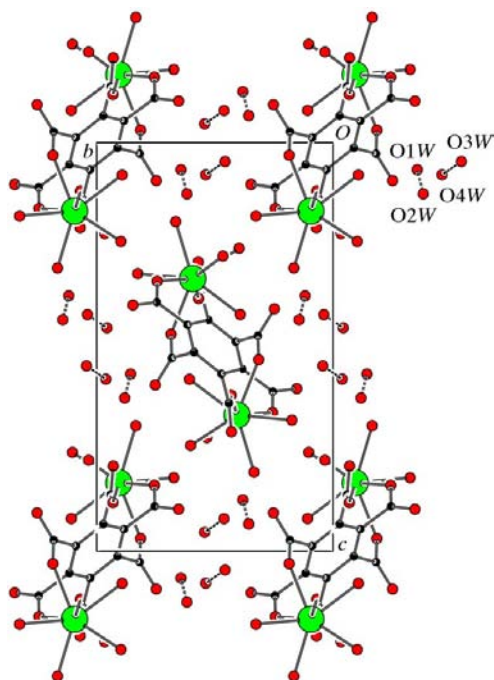
Figure 2
A projection view of (I) along the *a* axis, showing the packing of the infinite chains and the location of the crystallization water molecules, together with the labelling scheme for the water molecules.

(the twist angles lie in the range 50–70°). This twisting ability of a carboxylate group involved in a rare-earth-containing coordination polymer has already been reported in both a terephthalate compound (Reineke *et al.*, 1999) and a benzenetetracarboxylate compound (Cao *et al.*, 2002).

In addition to the five water molecules in the first coordination sphere of the Er atoms, two water molecules per Er atom were refined (Fig. 2). This is in accordance with the results of a thermogravimetric analysis. For the two sets of sites O1*W*–O4*W* and O2*W*–O3*W*, a disorder model (0.5:0.5) was introduced. These water molecules build a complex network of hydrogen bonding between themselves, the carboxylate O atoms and the water molecules of the first coordination sphere. The O atoms from the water molecules of

Figure 3
A projection view of the coordination polyhedron of the Er^{III} ions in (I), along with the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

crystallization exhibit high displacement parameters. We attribute these high parameters to the existence of numerous hydrogen bonds of comparable importance.

It is interesting to note that the crystal structure of (I) differs from the La-containing structure already reported by Wu *et al.* (1996). The La-containing material was synthesized by hydrothermal methods, whereas both Y- and Er-containing compounds have been obtained by slow diffusion in gel media.

Experimental

Mellitic acid was supplied by Aldrich Chemical Company Inc. and the oxide of erbium was supplied by Rhodia Inc. Both were used without further purification. By reaction with NaOH, mellitic acid provided the hexasodium mellitate salt. By reaction with HCl, the erbium oxide gave the pentahydrated erbium chloride. Dilute aqueous solutions of Er^{III} chloride and hexasodium mellitate were allowed to diffuse slowly through an agar gel medium in a U-shaped tube. After one and a half months, pale-pink single crystals of (I) were collected. Analysis calculated (found) for [Er$_2${C(CO$_2$)$_6$}(H$_2$O)$_{10}$]·4H$_2$O: Er 36.3 (36.3), C 15.5 (15.6), H 3.2 (3.0), O 45.3% (45.1%).

Crystal data

[Er$_2$(C$_{12}$O$_{12}$)(H$_2$O)$_{10}$]·4H$_2$O	$D_x = 2.444$ Mg m^{-3}
$M_r = 922.86$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 8641
$a = 8.4499 (1)$ Å	reflections
$b = 9.2595 (2)$ Å	$\theta = 1.0–30.0°$
$c = 16.2911 (4)$ Å	$\mu = 6.76$ mm^{-1}
$\beta = 100.3390 (8)°$	$T = 298 (2)$ K
$V = 1253.95 (4)$ Å^3	Plate, pale pink
$Z = 2$	$0.31 \times 0.09 \times 0.06$ mm

Data collection

Nonius KappaCCD area-detector diffractometer	3648 independent reflections
	2884 reflections with $I > 2\sigma(I)$
CCD rotation image scans, thick slices	$R_{int} = 0.049$
	$\theta_{max} = 30°$
Absorption correction: analytical (Alcock, 1970)	$h = -11 \rightarrow 9$
	$k = -13 \rightarrow 11$
$T_{min} = 0.256$, $T_{max} = 0.646$	$l = -21 \rightarrow 22$
17 561 measured reflections	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o{}^2) + (0.0438P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.037$	$+ 4.4981P]$
$wR(F^2) = 0.093$	where $P = (F_o{}^2 + 2F_c{}^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
3648 reflections	$\Delta\rho_{max} = 2.48$ e Å^{-3}
199 parameters	$\Delta\rho_{min} = -2.05$ e Å^{-3}
H-atom parameters not defined	

The s.u. values of the cell constants indicate the internal consistency of the measurements themselves, *i.e.* the precision of the measurements, not their accuracy. The maximum peak in the final

Table 1
Selected interatomic distances (Å).

Er—O31^i	2.281 (3)	Er—O1	2.351 (4)
Er—O5	2.341 (5)	Er—O3	2.358 (4)
Er—O21	2.343 (3)	Er—O4	2.362 (5)
Er—O11	2.350 (3)	Er—O2	2.391 (4)

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

difference map is 2.08 Å from O3W and the minimum peak is 0.77 Å from Er.

Data collection: *COLLECT* (Nonius, 1997–2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2000); software used to prepare material for publication: *SHELXL*97.

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