Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Poly[triaquatris(μ-4-pyridine-3,5-dicarboxylato)-didysprosium(III)]

Feng Li

The College of Chemistry, Xiang Tan University, Xiangtan, HuNan 411105, People's Republic of China

Correspondence e-mail: fengli@xtu.edu.cn

Received 30 October 2006
Accepted 28 November 2006

Key indicators

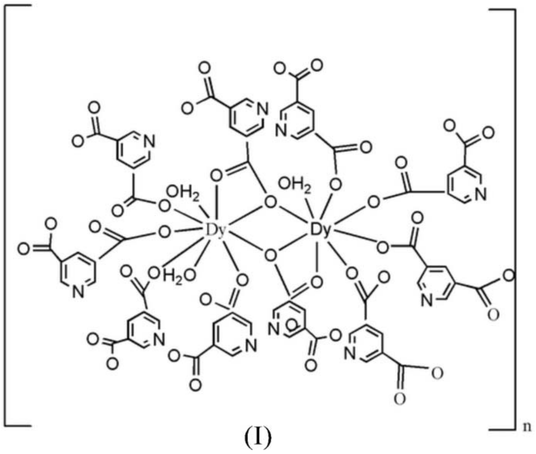
Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.009 Å
H-atom completeness 61%
R factor = 0.036
wR factor = 0.110
Data-to-parameter ratio = 11.8

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

The title complex, $[Dy_2(C_7H_3NO_4)_3(H_2O)_3]_n$, contains two independent dysprosium centers which are connected through two carboxylate O atoms into a binuclear unit. Each binuclear unit is linked to symmetry-related units by four 4-pyridine-3,5-dicarboxylate ligands to form a two-dimensional layer structure parallel to the *ac* plane. These two-dimensional layers are, in turn, linked into a three-dimensional network *via* shared carboxylate O atoms.

Comment

Much attention has been focused on the synthesis of zeolite-like materials with open-framework structures due to their potential applications in sorption, separation and heterogeneous catalysis (Li *et al.*, 1999). To synthesize such materials, rigid ligands such as 1,3,5-benzenetricarboxylate (BTC; Chui *et al.*, 1999) and 1,3-benzenedicarboxylate (BDC; Eddaoudi *et al.*, 2002) have been investigated widely, and many interesting complexes have been reported. Thus, modification of BTC and/or BDC ligands may yield more interesting results. 3,5-Pyridinedicarboxylate (H_2PDC) has three coordination sites, at the 1-, 3- and 5-positions of the benzene ring. This is similar to the BTC ligand. However, the affinity of oxygen for lanthanides is greater than for nitrogen. Thus, when the metal ions are lanthanides, nitrogen does not participate in coordination, providing only functionality in the channels of the extended structures. For this reason, PDC is similar to BDC. In this paper, we report the crystal structure of the title complex, (I), which was synthesized by the reaction of dysprosium(III) nitrate with PDC under hydrothermal conditions.



(I)

Part of the polymeric structure of (I) is shown in Fig. 1. The title complex is very similar to the corresponding Tm complex (Si *et al.*, 2004). The asymmetric unit in (I) consists of two independent Dy^{III} atoms (Dy1 and Dy2), three PDC ligands

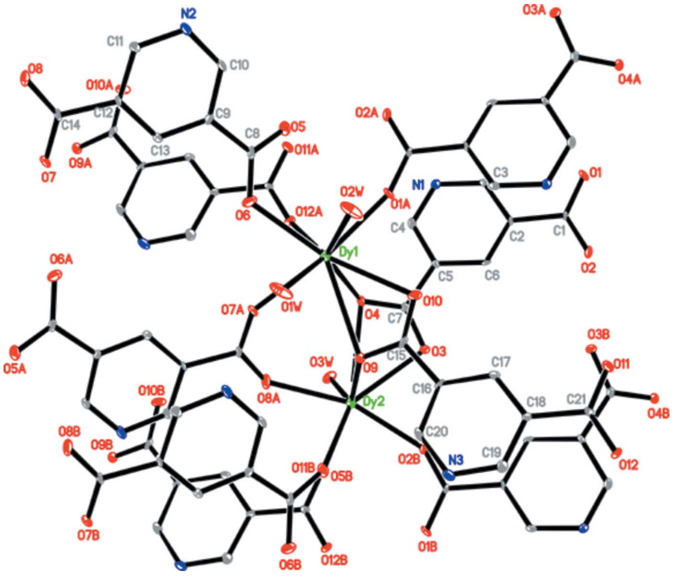


Figure 1
A portion of the polymeric structure of (I). H atoms have been omitted for clarity. Displacement ellipsoids are shown at the 30% probability level. [Symmetry codes: (*A*) $-x$, $-y + 1$, $-z$; (*B*) $-x + 1$, $-y + 1$, $-z$.]

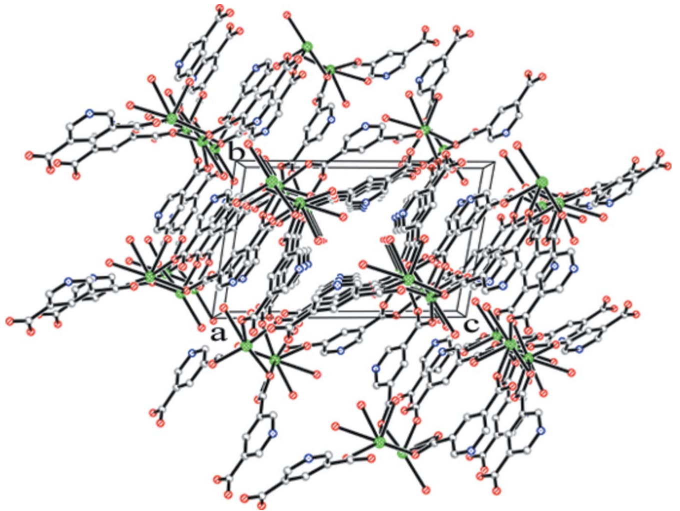


Figure 2
Partial packing plot of (I). H atoms have been omitted.

and three coordinated water molecules. Dy1 is coordinated by nine O atoms from six carboxylate groups and two water molecules, forming a square-face capped square antiprism. Dy2 is coordinated by seven O atoms from six carboxylate groups and one O atom from one water molecule, forming a 4,4′-bicapped trigonal prism. The bond lengths and angles in (I) are similar to those in the related structure of poly-[diaquadi-l5-succinato-l6-succinatodidysprosium(III)] (Wang *et al.*, 2006). By sharing two carboxylate O atoms *via* a monoatomic bridging mode, two Dy centers are connected into a binuclear unit. Each binuclear unit is briged to symmetry-related units by four PDC ligands to form a two-dimensional layer structure parallel to the *ac* plane. These two-dimensional layers, are in turn, linked into a three-dimensional network *via* shared carboxylate O atoms (Fig. 2).

Experimental

An aqueous NaOH solution (1 mol l^{-1}) was added to a mixture of $Dy(NO_3)\cdot6H_2O$ (1.50 mmol, 0.70 g) and H_2PDC (1.5 mmol, 0.25 g), to adjust the pH value to approximately 5. The resulting mixture was heated in a 23 ml stainless steel reactor with a Teflon liner at 423 K for three days. Colorless crystals were obtained in a yield of 35.6%.

Crystal data

$[Dy_2(C_7H_3NO_4)_3(H_2O)_3]$
$M_r = 874.36$
Triclinic, $P\bar{1}$
$a = 8.9266$ (2) Å
$b = 9.3595$ (1) Å
$c = 14.722$ (3) Å
$\alpha = 97.781$ (1)°
$\beta = 95.518$ (1)°
$\gamma = 104.819$ (1)°
$V = 1167.1$ (2) $Å^3$
$Z = 2$
$D_x = 2.488$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 6.44$ mm^{-1}
$T = 273$ (2) K
Needle, colorless
0.10 × 0.05 × 0.03 mm

Data collection

Bruker SMART CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.672$, $T_{max} = 0.826$
6372 measured reflections
4351 independent reflections
4055 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$
$\theta_{max} = 25.7°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.036$
$wR(F^2) = 0.110$
$S = 1.01$
4351 reflections
370 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0721P)^2 + 10.4697P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.24$ e $Å^{-3}$
$\Delta\rho_{min} = -2.03$ e $Å^{-3}$

H atoms of the coordinated water molecules were not located and neither were they included in the refinement. They are, however, included in the molecular formula. All other H atoms were positioned geometrically (C—H = 0.93 Å) and they were included in the refinement in the riding-model approximation with $U_{iso}(H) = 1.2U_{eq}(C)$. The highest peak in the final difference Fourier map is 1.00 Å from atom Dy1 and the deepest hole is 0.91 Å from atom Dy1.

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*.

References

Bruker (2004). *SMART* (Version 6.0), *SAINT* (Version 6.0) and *SHELXTL* (Version 5.1). Bruker AXS Inc., Madison, Wisconsin, USA.

Chui, S. S. Y., Lo, S. M. F., Charmant, J. P. H., Orpen, A. G. & Williams, I. D. (1999). *Science*, **283**, 1148–1150.

Eddaoudi, M., Jaheon Kim, J. B., Rosi, N., Vodak, D., Wachter, H. K., O'Keeffe, M. & Yaghi, O. M. (2002). *Science*, **295**, 469–472.

Li, H., Eddaoudi, M., O'Keeffe, M. & Yaghi, O. M. (1999). *Nature (London)*, **402**, 276–279.

Sheldrick, G. M. (1996). *SADABS*. University of Gottingen, Germany.

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

Si, S., Li, C., Wang, R. & Li, Y. (2004). *J. Coord. Chem.* **57**, 1545–1551.

Wang, C.-X., Li, Y., Zhang, Q.-H., Cai, D.-J. & Xie, X.-B. (2006). *Acta Cryst.* E**62**, m545–m547.