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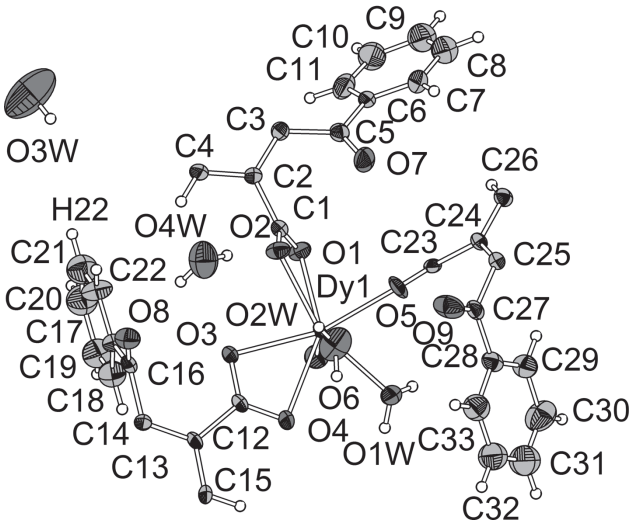
Crystal structure of *poly*-[tetraaqua-bis(μ_4-2,5-dibenzoyl-1,4-benzenedicarboxylato-κ^4 *O*^{1}:*O*^{2}:*O*^{3}:*O*^{4})-μ_2-2,5-dibenzoyl-1,4-benzenedicarboxylato-k^4 *O*^{5},*O*^{6}: *O*$^{5\prime}$,*O*$^{6\prime}$-didysprosium(III)] tetrahydrate $C_{33}H_{26}O_{13}Dy$



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Abstract

$C_{33}H_{26}O_{13}Dy$, triclinic, $P\bar{1}$ (no. 2), $a = 10.5417(4)$ Å, $b = 12.0923(6)$ Å, $c = 12.4320(4)$ Å, $\alpha = 82.373(3)°$, $\beta = 81.126(3)°$, $\gamma = 89.446(3)°$, $V = 1551.83(11)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0357$, $wR_{ref}(F^2) = 0.0820$, $T = 293$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block Size 0.30 × 0.28 × 0.27 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	24.8 cm^{-1}
Diffractometer, scan mode:	Xcalibur, ω
$2\theta_{max}$, completeness:	52.2°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12397, 6069, 0.035
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5479
$N(param)_{refined}$:	424
Programs:	Diamond [9], SHELX [10], CrysAlis^{PRO} [11]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Dy1	0.449728(18)	0.039262(16)	0.683732(15)	0.01835(8)
C1	0.4921(4)	0.2650(4)	0.5915(3)	0.0228(9)
C2	0.5016(4)	0.3865(4)	0.5445(3)	0.0215(9)
C3	0.5864(4)	0.4267(4)	0.4480(3)	0.0224(9)
C4	0.4167(4)	0.4606(4)	0.5947(3)	0.0237(10)
H4	0.3608	0.4342	0.6582	0.028*
C5	0.6893(4)	0.3511(4)	0.4013(4)	0.0248(10)
C6	0.7405(4)	0.3672(4)	0.2815(4)	0.0265(10)
C7	0.8668(5)	0.3376(4)	0.2484(5)	0.0402(13)
H7	0.9164	0.3072	0.3005	0.048*
C8	0.9193(6)	0.3531(5)	0.1376(5)	0.0506(15)
H8	1.0047	0.3355	0.1158	0.061*
C9	0.8462(6)	0.3935(5)	0.0623(5)	0.0551(16)
H9	0.8819	0.4038	−0.0116	0.066*
C10	0.7184(6)	0.4204(5)	0.0925(5)	0.0511(15)
H10	0.6683	0.4459	0.0391	0.061*
C11	0.6660(6)	0.4090(4)	0.2028(4)	0.0415(13)
H11	0.5814	0.4292	0.2240	0.050*

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Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C12	0.2302(4)	0.0183(4)	0.8383(4)	0.0279(11)
C13	0.1109(4)	0.0106(4)	0.9227(3)	0.0228(10)
C14	0.0441(4)	0.1063(4)	0.9490(3)	0.0223(9)
C15	0.0666(4)	−0.0939(4)	0.9736(4)	0.0269(10)
H15	0.1120	−0.1569	0.9557	0.032*
C16	0.0882(4)	0.2225(4)	0.8973(3)	0.0238(10)
C17	0.0327(4)	0.2703(4)	0.7992(4)	0.0271(10)
C18	−0.0521(6)	0.2093(5)	0.7539(5)	0.0478(14)
H18	−0.0752	0.1369	0.7860	0.057*
C19	−0.1018(6)	0.2547(5)	0.6624(5)	0.0550(16)
H19	−0.1580	0.2136	0.6321	0.066*
C20	−0.0667(7)	0.3639(6)	0.6154(6)	0.0629(18)
H20	−0.0998	0.3957	0.5534	0.076*
C21	0.0165(7)	0.4245(6)	0.6604(5)	0.0603(17)
H21	0.0398	0.4968	0.6283	0.072*
C22	0.0657(6)	0.3788(4)	0.7529(4)	0.0439(14)
H22	0.1206	0.4205	0.7840	0.053*
C23	0.7232(4)	0.0136(3)	0.5181(4)	0.0235(10)
C24	0.8664(4)	0.0038(4)	0.5095(3)	0.0206(9)
C25	0.9238(4)	−0.0428(4)	0.5988(3)	0.0217(9)
C26	0.9429(4)	0.0456(4)	0.4109(3)	0.0232(9)
H26	0.9045	0.0756	0.3512	0.028*
C27	0.8492(4)	−0.0964(4)	0.7066(4)	0.0250(10)
C28	0.7957(4)	−0.2099(4)	0.7103(4)	0.0290(11)
C29	0.8055(5)	−0.2650(5)	0.6182(4)	0.0402(13)
H29	0.8452	−0.2301	0.5504	0.048*
C30	0.7558(7)	−0.3726(5)	0.6279(6)	0.0605(18)
H30	0.7608	−0.4094	0.5663	0.073*
C31	0.6987(7)	−0.4245(6)	0.7298(6)	0.0637(18)
H31	0.6674	−0.4971	0.7365	0.076*
C32	0.6880(6)	−0.3712(5)	0.8191(5)	0.0538(16)
H32	0.6471	−0.4068	0.8863	0.065*
C33	0.7368(6)	−0.2642(5)	0.8128(5)	0.0463(14)
H33	0.7309	−0.2287	0.8753	0.056*
O1	0.4618(3)	0.1944(2)	0.5338(2)	0.0292(7)
O2	0.5070(3)	0.2369(3)	0.6899(2)	0.0292(7)
O3	0.2653(3)	0.1122(3)	0.7849(3)	0.0377(9)
O4	0.2938(3)	−0.0674(3)	0.8223(3)	0.0434(10)
O5	0.6621(3)	0.0236(3)	0.6112(3)	0.0352(8)
O6	0.3264(3)	−0.0132(3)	0.5669(3)	0.0359(8)
O7	0.7319(3)	0.2794(3)	0.4631(3)	0.0380(9)
O8	0.1578(3)	0.2757(3)	0.9410(3)	0.0360(8)
O9	0.8447(4)	−0.0520(3)	0.7886(3)	0.0491(10)
O1W	0.4923(3)	−0.1572(3)	0.6834(3)	0.0315(8)
H1WA	0.5170	−0.1725	0.6187	0.038*
H1WB	0.4366	−0.2062	0.7148	0.038*
O2W	0.5527(4)	0.0160(4)	0.8397(3)	0.0594(12)
H2WA	0.5253	−0.0393	0.8868	0.071*
H2WB	0.5672	0.0692	0.8747	0.071*
O3W	0.4250(8)	0.8399(5)	1.0045(6)	0.143(3)
H3WA	0.3729	0.8388	0.9588	0.172*
H3WB	0.4397	0.7732	1.0305	0.172*
O4W	0.4424(5)	0.3267(4)	0.8915(3)	0.0668(13)
H4WA	0.4671	0.3013	0.8312	0.080*
H4WB	0.3621	0.3144	0.9093	0.080*

Source of material

456 mg of $Dy(NO_3)_3{\cdot}6H_2O$ (0.1 mmol) and 56.10 mg of H_2L (H_2L = 2,5-dibenzoyl-1,4-benzenedicarboxylic acid, 0.15 mmol) were added in 15 mL of H_2O and the pH of system was adjusted to about 5.5 with 0.1 mol/L NaOH. The mixture was sealed in a 25 mL Teflon-lined reactor and heated at 423 K for 72 h. After cooled to room temperature at a rate of 3 °C·h^{-1}, colorless block crystals were collected as a pure phase. FT-IR (KBr, cm^{-1}): 3443(m), 3058(w), 1673(s), 1648(s), 1579(m), 1481(m), 1399(s), 1325(s), 1176(w), 1135(w), 1104(w), 1031(w), 1011(w), 931(w), 892(w), 807(w), 740(m), 620(m), 476(w).

Experimental details

The carbon H-atoms were placed in calculated positions (C—H_{phenyl} = 0.93 Å), and were included with $U_{iso}(H) = 1.2U_{eq}(C)$. The water H-atoms were placed in calculated positions (O—H = 0.85 Å), and were included with $U_{iso}(H) = 1.2U_{eq}(O)$.

Discussion

Some metal-organic frameworks (MOFs) are very promising multifunctional luminescent materials [1]. Lots of works have been concentrated on designing and synthesizing lanthanide-MOFs on aromatic ligands [2]. The bridging carboxylate ligands, such as 1,3/1,4-benzenedicarboxylate [3, 4] as well as modified 1,3/1,4-benzenedicarboxylate [5, 6], have frequently being utilized to produce polymeric structures. In addition, the structural sophistication can be further enhanced through the use of ancillary co-ligands. The use of 2,5-dibenzoyl-1,4-benzenedicarboxylic acid (H_2L) as educt for the synthesis of MOFs has recently been reported [7, 8]. The asymmetric unit of titled compound consists of one crystallographically independent Dy(III) ions, one and a half *L* anions, two coordinated and two lattice water molecules (*cf*. the figure). The Dy1 is coordinated by six carboxylate oxygen atoms from three *L* ligands (O1, O2, O3, O4, O5, O6) and two water ligands (O1W, O2W). The Dy—O bond lengths range from 2.246(3) to 2.485(3) Å (the average length is 2.3811 Å), and O—Dy—O bond angles are in the range 52.76(10)–158.09(14)°, which are comparable to those reported earlier for other Dy(III) complexes. In the polymeric structure of title compound, all carboxylic groups are deprotonated and *L* ligands display two types of bridging modes: μ_4 and μ_2. Two Dy1 ions are linked together by a pair of carboxylate groups (O5 and O6) of two *L* ligands, resulting in a 1D chain with binuclear $[Dy_2(COO)_2]$ units (4.7469(3) Å Dy$\cdots$Dy distance). Four carboxylate groups from the *L* ligands acting as bidentate (A1:2.188(5) Å of O3 and O4) bridge two adjacent chains to generate a 2D plane, which is further coordinated by the

carboxylate groups (A2:2.194(4) Å of O1 and O2) of *L* ligands to afford a 3D framework.

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