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A three-dimensional Eu(III) framework in the crystal structure of dimethylaminium poly [dimethylformamide- $\kappa^1 N$]bis(μ_4 -terephthalato- $\kappa^4 O:O':O'':O'''$)europium(III)] monohydrate, $C_{21}H_{25}EuN_2O_{10}$

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Abstract

$C_{21}H_{25}EuN_2O_{10}$, monoclinic, $P2_1/n$ (no. 14), $a = 14.1635(8)$ Å, $b = 12.1381(6)$ Å, $c = 14.4528(7)$ Å, $\beta = 104.361(2)^\circ$, $V = 2407.1(2)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0304$, $wR_{ref}(F^2) = 0.0893$, $T = 296$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The title complex was synthesized from a mixture of $Eu(NO_3)_3 \cdot 6H_2O$ (0.05 mmol), terephthalic acid (0.025 mmol)

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Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	$0.12 \times 0.10 \times 0.10$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	2.66 mm^{-1}
Diffractometer, scan mode:	BRUKER D8 VENTURE, φ and ω
$\theta_{\max}$, completeness:	27.5° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	22250, 5540, 0.049
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 4996
$N(\text{param})_{\text{refined}}$:	311
Programs:	BRUKER [1], OLEX2 [2], SHELX [3, 4]

and N,N -dimethylformamide (DMF) and H_2O mixed solution (4 mL, 3:1) sealed in a Teflon-lined reactor and heated at 115°C for 72 h. After the reactor was slowly cooled to room temperature, colorless block crystals were obtained and washed with DMF and ethanol successively, and then dried at room temperature.

Experimental details

All the H atoms on the benzene rings were placed geometrically.

Comment

Carboxyl ligands have been widely used to construct metal coordination polymers [5–7]. 1,4-Dicarboxybenzene and terephthalate are intriguing due to the ability to give rise to coordination polymers (CPs) that can be at the heart of synthesizing metal-organic framework (MOF) structures [8]. As far as we know, reports on Eu-containing CPs and molecular complexes are rare [9–11]. Recently, our group synthesized a related dinuclear Eu(III) complex [10]. Furthermore, different types of Eu(III) complexes will be formed due to the influence of different ligand coordination methods and synthesis conditions.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
Eu1	0.58824 (2)	0.44661 (2)	0.41149 (2)	0.01149 (7)
O1	0.1602 (2)	0.0904 (2)	0.08142 (17)	0.0159 (5)
O2	0.0518 (2)	0.0596 (2)	0.16787 (19)	0.0202 (6)
O3	0.3241 (2)	0.3784 (2)	0.53380 (18)	0.0167 (5)
O4	0.44669 (17)	0.3861 (2)	0.46597 (17)	0.0120 (5)
O5	0.53850 (19)	0.3712 (2)	0.23894 (17)	0.0137 (5)
O6	0.6150 (2)	0.5315 (2)	0.25567 (17)	0.0161 (5)
O7	0.75204 (19)	0.3961 (3)	0.4107 (2)	0.0201 (6)
O8	0.8456 (2)	0.3047 (2)	0.3355 (2)	0.0224 (6)
O9	0.5985 (2)	0.2368 (2)	0.4318 (2)	0.0217 (6)
N1	0.6446 (3)	0.1169 (3)	0.5548 (3)	0.0286 (8)
C1	0.1301 (3)	0.0968 (3)	0.1564 (2)	0.0120 (7)
C2	0.1946 (3)	0.1571 (3)	0.2396 (2)	0.0119 (7)
C3	0.2849 (3)	0.1963 (3)	0.2341 (2)	0.0147 (7)
H3	0.307820	0.181605	0.180376	0.018*
C4	0.3415 (3)	0.2575 (3)	0.3091 (3)	0.0150 (7)
H4	0.402208	0.283782	0.305485	0.018*
C5	0.3071 (2)	0.2794 (3)	0.3892 (2)	0.0108 (6)
C6	0.2171 (3)	0.2383 (3)	0.3947 (3)	0.0181 (8)
H6	0.194363	0.252348	0.448640	0.022*
C7	0.1613 (3)	0.1768 (3)	0.3209 (3)	0.0193 (8)
H7	0.101509	0.148683	0.325394	0.023*
C8	0.3629 (2)	0.3515 (3)	0.4680 (2)	0.0096 (6)
C9	0.5638 (3)	0.4601 (3)	0.2042 (2)	0.0121 (7)
C10	0.5303 (3)	0.4801 (3)	0.0984 (2)	0.0119 (7)
C11	0.4827 (3)	0.3982 (3)	0.0364 (2)	0.0162 (7)
H11	0.471049	0.329844	0.060399	0.019*
C12	0.5473 (3)	0.5817 (3)	0.0609 (3)	0.0159 (7)
H12	0.579201	0.636746	0.101619	0.019*
C13	0.8330 (3)	0.3751 (3)	0.3949 (2)	0.0118 (6)
C14	0.9204 (3)	0.4381 (3)	0.4507 (2)	0.0112 (7)
C15	0.9120 (3)	0.5061 (3)	0.5257 (3)	0.0152 (7)
H15	0.853251	0.509927	0.543228	0.018*
C16	1.0090 (3)	0.4317 (3)	0.4256 (3)	0.0155 (7)
H16	1.015300	0.385601	0.376044	0.019*
C17	0.5823 (3)	0.1845 (3)	0.4997 (3)	0.0251 (9)
H17	0.521781	0.193574	0.512887	0.030*
C18	0.6198 (5)	0.0561 (5)	0.6329 (5)	0.0532 (17)
H18A	0.662678	0.077833	0.692480	0.080*
H18B	0.626688	−0.021522	0.623438	0.080*
H18C	0.553649	0.071926	0.633933	0.080*
C19	0.7427 (4)	0.1039 (5)	0.5443 (4)	0.0441 (13)
H19A	0.748689	0.033874	0.515501	0.066*
H19B	0.787762	0.107527	0.606012	0.066*
H19C	0.756913	0.161763	0.504530	0.066*
O10	0.9297 (4)	0.3047 (5)	0.1765 (4)	0.0833 (19)
H10A	0.971987	0.262129	0.163116	0.125*
H10B	0.913257	0.291679	0.227706	0.125*
N2	0.6758 (3)	0.1940 (3)	0.2537 (3)	0.0239 (8)
H2A	0.627724	0.234695	0.265189	0.029*
H2B	0.731634	0.218916	0.290645	0.029*
C20	0.6621 (4)	0.0789 (4)	0.2790 (4)	0.0355 (11)
H20A	0.600372	0.052701	0.241669	0.053*
H20B	0.663482	0.074273	0.345596	0.053*
H20C	0.713427	0.034388	0.266111	0.053*

Table 2: (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}^*/U_{eq}
C21	0.6776 (4)	0.2069 (5)	0.1534 (3)	0.0352 (11)
H21A	0.615746	0.185162	0.113148	0.053*
H21B	0.728038	0.161334	0.140047	0.053*
H21C	0.690079	0.282528	0.141156	0.053*

As shown in Figure (part a), there is a crystallographically independent Eu^{3+} named Eu1. The adjacent Eu^{3+} is bridged by eight oxygen atoms from four terephthalato ligands and one DMF ligand to form a binuclear Eu_2 unit, where the distance between Eu^{3+} and Eu^{3+} in the binuclear Eu_2 unit is about 4.007 Å (see part b of the figure). In this structure, the average Eu–O bond length is about 2.532 Å. In addition, the adjacent dinuclear Eu_2 units are connected to each other by bridging terephthalato ligands to expand into a three-dimensional framework structure. The voids of the structure are filled with dimethylammonium cations and water molecules, and the dimethylamine cations play a role in balancing the charge.

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