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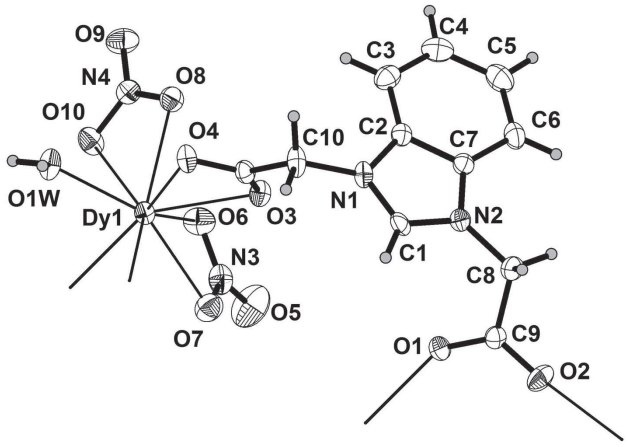
Crystal structure of poly[aqua-bis(nitrato-κ^2*O*,*O*′)-(μ$_3$-1,3-benzimidazol-3-ium-1,3-diacetato-κ^4*O*,*O*′:*O*″:*O*‴)dysprosium(III)], $C_{11}H_{11}DyN_4O_{11}$



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Abstract

$C_{11}H_{11}DyN_4O_{11}$, monoclinic, $P2_1/c$ (no. 14), $a = 12.073(4)$ Å, $b = 14.704(4)$ Å, $c = 8.763(3)$ Å, $\beta = 94.233(5)°$, $V = 1551.3(8)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0201$, $wR_{ref}(F^2) = 0.0555$, $T = 293$ 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:	0.18 × 0.18 × 0.12 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	48.9 cm^{-1}
Diffractometer, scan mode:	Bruker P4, ω scans
$2\theta_{max}$, completeness:	54°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9235, 3379, 0.018
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2994
$N(param)_{refined}$:	244
Programs:	Bruker programs [1], SHELX [2]

Source of material

1-(Carboxymethyl)-1*H*-benzimidazol-3-ium-3-acetate was prepared according to the reported procedures [3]. Sodium carbonate (0.25 mmol) was added to 1-(carboxymethyl)-1*H*-benzimidazol-3-ium-3-acetate (0.5 mmol) in distilled water (5 mL) while stirring at room temperature. Once the solution became clear, dysprosium nitrate (0.25 mmol) dissolved in distilled water (5 mL) was added dropwise. After continuous stirring for 15 min, the resulting solution was filtered and placed undisturbed for 2 weeks. Colourless crystals suitable for crystal structure analysis were obtained.

Experimental details

Hydrogen atoms bound to carbon were placed in geometrically idealized positions and refined isotropic using riding model with $U_{iso}(H) = 1.2U_{eq}(C)$. The hydrogens bound to coordinated water molecules were located in difference Fourier map and refined with $U_{iso}(H) = 1.2U_{eq}(O)$.

Comment

In the recent years, the benzimidazole derivatives and their metal complexes have attracted much attention because of the specific topological frameworks and intresting properties of materials [4–7]. Furthermore, benzimidazole carboxylic acids and their derivatives are widely used due to their biological activites such as antifilarial, antineoplastic, anthelmintic and antiviral activities [8–11]. Up to now, several complexes

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

Atom	x	y	z	U_{iso}*/U_{eq}
Dy1	0.827724(10)	0.622154(7)	0.153115(12)	0.01925(6)
O1	1.03690(16)	0.30317(13)	−0.3104(2)	0.0297(4)
O2	1.07952(17)	0.19565(14)	−0.4752(2)	0.0348(5)
O3	0.78840(16)	0.49057(12)	−0.0147(2)	0.0259(4)
O4	0.71253(19)	0.62053(11)	−0.0928(2)	0.0271(4)
O5	1.0187(3)	0.4143(2)	0.3482(3)	0.0701(9)
O6	0.86727(19)	0.49409(15)	0.3255(2)	0.0385(5)
O7	0.99773(19)	0.52234(14)	0.1794(2)	0.0386(5)
O8	0.64434(17)	0.56117(13)	0.2082(2)	0.0293(4)
O9	0.56479(18)	0.57656(16)	0.4217(2)	0.0405(5)
O10	0.7271(2)	0.63656(14)	0.3932(2)	0.0333(5)
N1	0.7357(2)	0.40731(15)	−0.2986(2)	0.0240(5)
N2	0.81350(19)	0.28621(15)	−0.3867(3)	0.0254(5)
N3	0.9632(2)	0.47502(17)	0.2852(3)	0.0355(6)
N4	0.6424(2)	0.59069(17)	0.3431(3)	0.0268(5)
C1	0.8143(2)	0.37567(17)	−0.3810(3)	0.0248(6)
H1A	0.8641	0.4121	−0.4293	0.030*
C2	0.6788(2)	0.33277(17)	−0.2420(3)	0.0223(5)
C3	0.5908(2)	0.3274(2)	−0.1509(3)	0.0314(6)
H3A	0.5585	0.3793	−0.1127	0.038*
C4	0.5530(3)	0.2416(2)	−0.1193(4)	0.0391(7)
H4A	0.4939	0.2352	−0.0578	0.047*
C5	0.6016(3)	0.1640(2)	−0.1776(4)	0.0383(7)
H5A	0.5735	0.1072	−0.1539	0.046*
C6	0.6898(2)	0.16848(18)	−0.2689(3)	0.0299(6)
H6A	0.7213	0.1165	−0.3079	0.036*
C7	0.7289(2)	0.25542(17)	−0.2995(3)	0.0228(5)
C8	0.8921(2)	0.2306(2)	−0.4662(3)	0.0312(6)
H8A	0.8831	0.2443	−0.5747	0.037*
H8B	0.8736	0.1670	−0.4534	0.037*
C9	1.0131(2)	0.24475(17)	−0.4109(3)	0.0225(5)
C10	0.7089(3)	0.50271(17)	−0.2752(3)	0.0305(7)
H10A	0.6298	0.5110	−0.2989	0.037*
H10B	0.7471	0.5390	−0.3473	0.037*
C11	0.7386(2)	0.53891(17)	−0.1157(3)	0.0227(5)
O1W	0.73540(18)	0.76199(12)	0.1590(2)	0.0304(4)
H1WB	0.7278	0.7950	0.0777	0.037*
H1WA	0.7348	0.7964	0.2410	0.037*

have been built by benzimidazole carboxylic acid [12–17]. To extend this work, a crystal structure of a related samarium complex was reported by us [18].

The asymmetric unit of the title structures consists of one Dy^{3+} cation, one 1-(carboxymethyl)-1,3-benzimidazol-3-ium-3-acetate ligand, two nitrate ions and one water molecule. The Dy atom is nine-coordinated showing a distorted, tri-capped trigonal prism. The coordination sphere is composed of nine oxygen atoms from a chelating carboxylate group, two monodentate coordinating carboxylate groups, two chelating nitrato ligands, and one coordinated water molecule. The sets of atoms O1w, O8, O10 and O2^{i} ($i = x+2,\ y+1/2,\ -z-1/2$), O3, O7 define the triangular base and upperface, respectively, and O1^{ii} (ii $= -x+2,\ -y+1,\ -z$), O4, O6 achieve the cappings of the polyhedron. The Dy—O bond lengths range from 2.3333(19) Å to 2.2608(18) Å. Meanwhile, the two carboxylate groups of one ligand take a μ_3-coordination mode bridging three Dy^{3+} ions to form a two dimensional undulating sheet structure.

Furthermore, there are neither obvious π–π stacking interactions, nor hydrogen bonds existing between the adjacent sheets. Therefore, the packing seems to be dominated by van der Waals forces. To simplify the structure of the two dimensional framework, the Dy atom and the ligand both can be viewed as 3-connected node, therefore the topology of the resulting structure may be described as fes topology with a point symbol of (4.8^2) [19].

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