Crystal structure of poly[aqua-bis(nitrato-κ^2O,O')-(μ_3-1,3-benzimidazol-3-ium-1,3-diacetato-$\kappa^4O,O':O'':O'''$)samarium(III)], $C_{11}H_{11}N_4O_{11}Sm$

Qian Zhang[*, I], Li He[II] and Qiong-Yan Yu[III]

[I] School of Materials and Chemical Engineering, Henan Institute of Engineering, Zhengzhou 451191, Henan Province, P. R. China
[II] School of Computer Science, Henan Institute of Engineering, Zhengzhou 451191, Henan Province, P. R. China
[III] School of Science, Henan Institute of Engineering, Zhengzhou 451191, Henan Province, P. R. China

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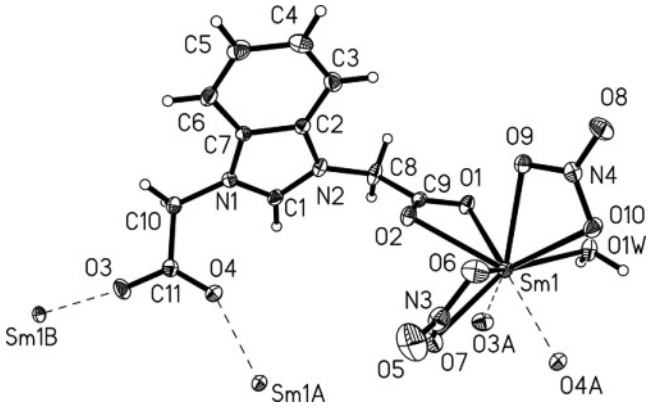

Abstract

$C_{11}H_{11}N_4O_{11}Sm$, monoclinic, $P2_1/c$ (no. 14), a = 12.091(4) Å, b = 14.756(5) Å, c = 8.839(3) Å, β = 94.242(6)°, V = 1572.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.0258, $wR_{ref}(F^2)$ = 0.0586, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.31×0.34×0.41 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	38.07 cm^{-1}
Diffractometer, scan mode:	Bruker P4, ω
$2\theta_{max}$:	54.16°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	9309, 3444
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2837
$N(param)_{refined}$:	244
Programs:	SHELX [18]

Source of material

The ligand 1-(carboxymethyl)-1*H*-benzimidazol-3-ium-3-acetate was prepared according to the reported procedures [1]. Solid 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, samarium nitrate hexahydrate (0.25 mmol) in distilled water (5 mL) was added dropwise. After continuous stirring for 15 min., the resulting blue solution was filtered and placed undisturbed for 2 weeks. During this period, diffusion slowly took place, and coloourless crystals suitable for X-ray 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).

Discussion

In the recent years, the benzimidazole derivatives and their metal complexes has attracted much attention because of the specific topological frameworks and interesting properties of materials [2–5]. Furthmore, benzimidazole carboxylic acids and their derivatives are widely used due to their biological activites such as antifilarial, antineoplastic, anthelmintic and antiviral activities [6–9]. Up to now, several complexes have been synthesized using benzimidazole carboxylates [10–16]. To extend this research, we used a benzimidazole carboxylic acid, 1,3-benzimidazol-3-ium-1,3-diacetic acid to react with Sm^{3+} ions. The asymmetric unit of the title structures consists of one Sm^{3+} cation, one 1-(carboxymethyl)-1,3-benzimidazol-3-ium-3-acetate ligand, two nitrate ions and one water molecule. The Sm atom is nine-coordinated with a distorted, tricapped trigonal prism by nine oxygen atoms from a chelating carboxylate group, two monodentate carboxylate groups, two chelating nitrate ions, and one coordinated aqua molecule. The sets of atoms O1, O2, O3^{i} (i = –x+1, y+½, –z+½) and O4^{ii} (ii = –x+1, –y+1, –z+1), O6, O10 define the tiangular base and upperface, respectively, and O1w, O7, O9, achieve the cappings of the polyhedron. The Sm–O bond lengths range from 2.307(3) Å to 2.560(3) Å. The two carboxylate groups of each benzimidazole-3-ium-1,3-diacetato ligand take a (κ^1-κ^1-μ_2)-(κ^2)-μ_3 coordination mode bridging three Sm^{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 Sm atom and the ligand both can be viewed as 3-connected node, therefore the topology of the resulting structure may be described as a topology with a point symbol of (4.8^2) [17].

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1A)	4*e*	0.3589	0.4110	0.0693	0.031
H(3A)	4*e*	0.0551	0.3779	0.3872	0.039
H(4A)	4*e*	–0.0069	0.2342	0.4431	0.047
H(5A)	4*e*	0.0709	0.1074	0.3449	0.047
H(6A)	4*e*	0.2182	0.1176	0.1903	0.037
H(8A)	4*e*	0.1256	0.5097	0.2035	0.039
H(8B)	4*e*	0.2421	0.5372	0.1522	0.039
H(10A)	4*e*	0.3786	0.2452	–0.0743	0.039
H(10B)	4*e*	0.3682	0.1676	0.0445	0.039
H(1WA)	4*e*	0.2555	0.7994	0.5729	0.038
H(1WB)	4*e*	0.2616	0.7950	0.7506	0.038

* Correspondence author (e-mail: diamond_zq@126.com)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sm(1)	4*e*	0.33159(2)	0.62032(1)	0.65213(2)	0.0239(1)	0.0193(1)	0.01787(9)	–0.00325(8)	0.00334(6)	–0.00016(7)
N(1)	4*e*	0.3080(3)	0.2861(2)	0.1119(3)	0.022(2)	0.026(2)	0.027(2)	0.001(1)	0.001(1)	–0.005(1)
N(2)	4*e*	0.2302(3)	0.4068(2)	0.2003(3)	0.034(2)	0.018(1)	0.022(2)	0.001(1)	–0.003(1)	–0.002(1)
N(3)	4*e*	0.4670(3)	0.4707(2)	0.7891(4)	0.047(2)	0.030(2)	0.036(2)	0.002(2)	–0.005(2)	0.000(2)
N(4)	4*e*	0.1437(3)	0.5901(2)	0.8441(3)	0.031(2)	0.026(2)	0.024(2)	0.003(1)	0.005(2)	0.004(1)
O(1)	4*e*	0.2146(2)	0.6189(2)	0.4056(3)	0.037(2)	0.019(1)	0.027(1)	0.000(1)	–0.001(1)	–0.002(1)
O(2)	4*e*	0.2855(2)	0.4873(2)	0.4821(3)	0.037(2)	0.022(1)	0.021(1)	0.002(1)	–0.001(1)	–0.001(1)
O(3)	4*e*	0.5735(2)	0.1954(2)	0.0270(3)	0.033(2)	0.043(2)	0.030(2)	0.009(1)	0.008(1)	–0.008(1)
O(4)	4*e*	0.5303(2)	0.3032(2)	0.1888(3)	0.027(2)	0.032(2)	0.032(2)	–0.001(1)	–0.002(1)	–0.010(1)
O(5)	4*e*	0.5224(4)	0.4115(3)	0.8542(5)	0.086(3)	0.061(2)	0.076(3)	0.040(2)	–0.004(2)	0.023(2)
O(6)	4*e*	0.3696(3)	0.4887(2)	0.8256(3)	0.040(2)	0.046(2)	0.042(2)	–0.003(2)	0.003(1)	0.017(1)
O(7)	4*e*	0.5024(3)	0.5176(2)	0.6848(3)	0.040(2)	0.038(2)	0.048(2)	0.007(1)	0.011(1)	0.004(1)
O(8)	4*e*	0.0661(3)	0.5764(2)	0.9218(3)	0.036(2)	0.055(2)	0.035(2)	–0.001(2)	0.016(1)	0.007(1)
O(9)	4*e*	0.1443(2)	0.5601(2)	0.7106(3)	0.030(2)	0.036(2)	0.026(1)	–0.003(1)	0.004(1)	–0.007(1)
O(10)	4*e*	0.2287(2)	0.6350(2)	0.8938(3)	0.039(2)	0.039(2)	0.025(1)	–0.012(1)	0.007(1)	–0.007(1)
C(1)	4*e*	0.3091(3)	0.3749(2)	0.1173(4)	0.029(2)	0.027(2)	0.022(2)	–0.007(2)	0.007(1)	–0.005(2)
C(2)	4*e*	0.1749(3)	0.3321(2)	0.2564(4)	0.023(2)	0.021(2)	0.022(2)	0.001(1)	–0.004(1)	0.000(1)
C(3)	4*e*	0.0875(3)	0.3264(3)	0.3488(4)	0.030(2)	0.037(2)	0.029(2)	0.004(2)	0.001(2)	–0.005(2)
C(4)	4*e*	0.0511(4)	0.2410(3)	0.3805(5)	0.030(2)	0.048(3)	0.041(2)	–0.004(2)	0.008(2)	0.007(2)
C(5)	4*e*	0.0988(4)	0.1641(3)	0.3216(5)	0.040(3)	0.030(2)	0.048(3)	–0.006(2)	0.001(2)	0.010(2)
C(6)	4*e*	0.1864(3)	0.1691(2)	0.2295(4)	0.035(2)	0.020(2)	0.036(2)	–0.003(2)	0.000(2)	–0.002(2)
C(7)	4*e*	0.2237(3)	0.2555(2)	0.1992(4)	0.018(2)	0.025(2)	0.022(2)	0.001(1)	–0.004(1)	–0.003(1)
C(8)	4*e*	0.2048(4)	0.5010(2)	0.2246(4)	0.052(3)	0.019(2)	0.026(2)	0.005(2)	–0.007(2)	–0.003(1)
C(9)	4*e*	0.2376(3)	0.5364(2)	0.3834(4)	0.024(2)	0.022(2)	0.022(2)	–0.003(2)	0.002(2)	–0.003(1)
C(10)	4*e*	0.3868(3)	0.2310(3)	0.0331(4)	0.030(2)	0.036(2)	0.032(2)	–0.001(2)	0.005(2)	–0.016(2)
C(11)	4*e*	0.5074(3)	0.2451(2)	0.0902(4)	0.028(2)	0.026(2)	0.021(2)	0.000(2)	0.004(2)	0.000(1)
O(1W)	4*e*	0.2377(2)	0.7637(2)	0.6586(3)	0.047(2)	0.024(1)	0.025(1)	0.004(1)	0.005(1)	–0.000(1)

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