The pseudosymmetric crystal structure of a Dy(III) complex with 3-(3-pyridin-4-yl-[1,2,4]oxadiazol-5-yl)propionic acid, $C_{30}H_{26}DyN_9O_{10}$

Fang Zhang[I], Fang Huang[I], Ying Jin[I], Qifan Chen[II], Wenjing Quan[III] and Fei Liu[*, I]

[I] College of Chemical Engineering & Materials, Eastern Liaoning University; Dandong 118003, Liaoning Province, P. R. China
[II] Experiment Center, Eastern Liaoning University, Dandong 118003, Liaoning Province, P. R. China
[III] Feng Cheng Environmental Monitoring Station, Dandong 118100, Liaoning Province, P. R. China

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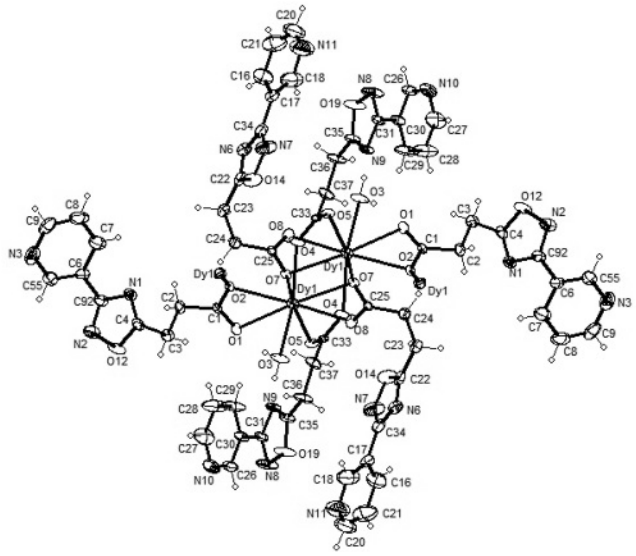

Abstract

$C_{30}H_{26}DyN_9O_{10}$, triclinic, $P\bar{1}$ (no. 2), a = 7.706(5) Å, b = 14.965(5) Å, c = 15.859(5) Å, α = 115.358(5)°, β = 93.621(5)°, γ = 101.313(5)°, V = 1598.0 Å^3, Z = 2, $R_{gt}(F)$ = 0.0417, $wR_{ref}(F^2)$ = 0.1248, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.13×0.21×0.3 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	24.11 cm^{-1}
Diffractometer, scan mode:	Xcalibur, Eos, φ and ω
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10694, 5634
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5205
$N(param)_{refined}$:	451
Programs:	SHELX [7]

Source of material

3-(3-Pyridin-4-yl-[1,2,4]oxadiazol-5-yl)-propionic acid (*HL*) is easily available by a literature known synthesis [1]. A solution of ammonia (0.5 M) was added dropwise to a methanol (15 mL) solution of *HL* (3 mmol), resulting in a transparent solution. A methanol (15 mL) solution of Dy(III) nitrate hexahydrate (1 mmol) was added into the resulting solution and allowed to stir for 12 h. The suspension was filtered and diethyl ether was allowed to diffuse slowly into the solution of the filtrate. Colourless crystals were obtained in about 3 weeks.

* Correspondence author (e-mail: berylliu8090@sina.com)

Experimental details

H atoms bound to C atoms were placed in calculated positions and treated as riding on their parent atoms, with C–H = 0.93 or 0.97 Å and U_{iso}(H) = 1.2 or 1.5 U_{eq}(C). H atom of the hydroxy groups and water molecules were refined with restraint an O–H distance of 0.85 Å

Discussion

Lanthanide-based carboxylate complexes have been found to exhibit anticancer, unusual coordination characteristics, exceptional optical, magnetic properties [2–4] precursors for oxides [5] and fungicidal properties [6]. As an ongoing part of our investigations, we report the preparation and crystal structure of the title compound. In the molecular structure of the title complex, the coordination sphere of each Dy^{III} ion consists of one water ligand and eight oxygen atoms from three ligands to form a nine coordination (see Fig). Three ligands are of two coordination modes with one belonging to mode A and the rest two in mode B. Mode A: O4 and O5 of the carboxylic group are connected to two adjacent Dy^{III} ions, respectively. Mode B: O1 and O2 of the carboxylic group are connected to the same Dy^{III} ion, O2 is also connected to adjacent Dy^{III} ion. The title molecular structure is built up of ligands that bridge two pairs of such Dy^{III} ions through the deprotonated carboxylate oxygen atoms. The Dy^{III} ions are bridged up by two O2-atoms and two O7-atoms with Dy1···Dy1 distance of 4.2631(2) and 4.1193(2) Å, respectively. The dihedral angles between Dy1–O2–Dy1–O2 and O2–C1–O1–Dy1 as well as Dy1–O7–Dy1–O7 and Dy1–O5–Dy1–O5 are 40.405(2)° and 87.780(1)°, respectively. The bond distances of Dy–O are in the range of 2.471(4)–2.647(4) Å, (ave. 2.551 Å). A coordination polymer oriented along the *a* direction is present.

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

Atom	Site	x	y	z	U_{iso}
H(2A)	2*i*	–0.0767	0.1871	0.7380	0.026
H(2B)	2*i*	0.1089	0.2600	0.7495	0.026
H(3A)	2*i*	0.0048	0.1673	0.8638	0.032
H(3B)	2*i*	0.2070	0.1803	0.8513	0.032
H(7)	2*i*	0.3900	0.5661	0.9188	0.049
H(8)	2*i*	0.4923	0.7399	0.9675	0.057
H(9)	2*i*	0.4527	0.8517	1.1117	0.054
H(16)	2*i*	0.8721	0.5774	0.5014	0.055
H(18)	2*i*	0.5685	0.3419	0.2735	0.086
H(20)	2*i*	0.9028	0.5493	0.2438	0.084
H(21)	2*i*	0.9955	0.6417	0.4021	0.077
H(23A)	2*i*	0.4986	0.4266	0.6992	0.036
H(23B)	2*i*	0.6867	0.4015	0.6987	0.036
H(24A)	2*i*	0.4643	0.2801	0.7180	0.029

Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(24B)	2*i*	0.3600	0.2487	0.6175	0.029
H(26)	2*i*	0.1635	–0.0454	–0.1963	0.034
H(27)	2*i*	–0.1216	–0.3260	–0.3333	0.060
H(28)	2*i*	–0.1092	–0.3439	–0.1966	0.076
H(29)	2*i*	0.0460	–0.2108	–0.0581	0.063
H(36A)	2*i*	0.4139	0.1641	0.2544	0.040

Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(36B)	2*i*	0.5322	0.0848	0.2274	0.040
H(37A)	2*i*	0.2746	–0.0367	0.2242	0.033
H(37B)	2*i*	0.1841	0.0541	0.2695	0.033
H(55)	2*i*	0.2021	0.6331	1.1599	0.040
H(101)	2*i*	0.0048	–0.1754	0.6023	0.036
H(102)	2*i*	–0.0939	–0.1442	0.5498	0.036

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}
C(1)	2*i*	0.1049(7)	0.1106(4)	0.6742(4)	0.011(2)	0.011(2)	0.010(3)	–0.004(2)	0.002(2)	0.001(2)
C(2)	2*i*	0.0522(8)	0.1969(4)	0.7509(4)	0.028(3)	0.020(3)	0.011(3)	0.007(2)	0.002(2)	0.002(2)
C(3)	2*i*	0.103(1)	0.2074(5)	0.8498(4)	0.046(4)	0.017(3)	0.009(3)	0.004(3)	0.001(3)	0.001(2)
C(4)	2*i*	0.1442(9)	0.3154(5)	0.9238(4)	0.035(3)	0.021(3)	0.008(3)	0.003(3)	0.003(2)	0.002(2)
C(6)	2*i*	0.2851(8)	0.5813(5)	1.0361(4)	0.027(3)	0.019(3)	0.020(3)	–0.002(2)	–0.003(2)	0.008(3)
C(7)	2*i*	0.373(1)	0.6132(6)	0.9772(5)	0.059(5)	0.031(4)	0.028(4)	0.001(3)	0.009(3)	0.013(3)
C(8)	2*i*	0.434(1)	0.7161(6)	1.0062(6)	0.059(5)	0.036(4)	0.054(5)	0.000(4)	0.011(4)	0.030(4)
C(9)	2*i*	0.408(1)	0.7823(6)	1.0926(7)	0.038(4)	0.022(4)	0.071(6)	0.005(3)	0.005(4)	0.020(4)
C(16)	2*i*	0.836(1)	0.5420(7)	0.4362(6)	0.036(4)	0.059(5)	0.043(5)	0.001(4)	0.002(3)	0.029(4)
C(17)	2*i*	0.709(1)	0.4510(5)	0.3968(5)	0.044(4)	0.027(3)	0.034(4)	0.015(3)	0.010(3)	0.018(3)
C(18)	2*i*	0.655(2)	0.4036(7)	0.3000(7)	0.13(1)	0.042(5)	0.041(5)	0.006(6)	0.010(6)	0.022(4)
C(20)	2*i*	0.848(2)	0.525(1)	0.2832(8)	0.076(7)	0.102(9)	0.077(8)	0.034(7)	0.039(6)	0.072(7)
C(21)	2*i*	0.908(1)	0.5803(8)	0.3777(9)	0.036(5)	0.071(6)	0.108(9)	0.002(4)	0.011(5)	0.065(7)
C(22)	2*i*	0.5747(9)	0.3798(5)	0.5706(5)	0.028(3)	0.018(3)	0.034(4)	0.010(3)	0.005(3)	0.009(3)
C(23)	2*i*	0.566(1)	0.3785(5)	0.6635(5)	0.043(4)	0.018(3)	0.033(4)	0.012(3)	0.013(3)	0.011(3)
C(24)	2*i*	0.4786(8)	0.2734(5)	0.6554(5)	0.025(3)	0.021(3)	0.031(3)	0.007(3)	0.012(3)	0.013(3)
C(25)	2*i*	0.5853(8)	0.1966(4)	0.6118(4)	0.021(3)	0.015(3)	0.010(3)	0.003(2)	0.005(2)	0.004(2)
C(26)	2*i*	0.1088(9)	–0.1065(5)	–0.1962(5)	0.029(3)	0.039(4)	0.029(4)	0.008(3)	0.007(3)	0.026(3)
C(27)	2*i*	–0.059(1)	–0.2713(7)	–0.2769(5)	0.063(5)	0.052(5)	0.022(4)	–0.001(4)	–0.007(4)	0.014(4)
C(28)	2*i*	–0.052(2)	–0.2825(7)	–0.1954(6)	0.103(8)	0.042(5)	0.033(4)	–0.020(5)	–0.009(5)	0.024(4)
C(29)	2*i*	0.039(1)	–0.2035(6)	–0.1135(5)	0.079(6)	0.048(5)	0.024(4)	–0.017(4)	–0.008(4)	0.027(4)
C(30)	2*i*	0.1225(9)	–0.1118(5)	–0.1114(4)	0.030(3)	0.034(3)	0.015(3)	0.004(3)	0.002(2)	0.016(3)
C(31)	2*i*	0.2240(9)	–0.0268(5)	–0.0226(4)	0.033(3)	0.032(3)	0.019(3)	0.007(3)	0.009(3)	0.018(3)
C(33)	2*i*	0.3799(7)	0.0560(4)	0.3627(4)	0.021(3)	0.016(3)	0.016(3)	0.005(2)	0.001(2)	0.012(2)
C(34)	2*i*	0.6257(9)	0.4059(5)	0.4546(5)	0.037(4)	0.022(3)	0.032(4)	0.012(3)	0.005(3)	0.011(3)
C(35)	2*i*	0.3390(9)	0.0536(5)	0.1190(4)	0.031(3)	0.032(3)	0.021(3)	0.001(3)	0.004(3)	0.020(3)
C(36)	2*i*	0.410(1)	0.0921(5)	0.2210(4)	0.044(4)	0.034(4)	0.015(3)	–0.013(3)	–0.004(3)	0.016(3)
C(37)	2*i*	0.2985(8)	0.0359(6)	0.2656(4)	0.021(3)	0.046(4)	0.019(3)	0.000(3)	0.000(2)	0.021(3)
C(55)	2*i*	0.2636(9)	0.6545(5)	1.1206(5)	0.030(4)	0.031(4)	0.032(4)	0.001(3)	0.005(3)	0.012(3)
C(92)	2*i*	0.2189(9)	0.4722(5)	1.0084(4)	0.035(4)	0.019(3)	0.015(3)	–0.003(3)	0.001(3)	0.006(2)
Dy(1)	2*i*	0.24099(4)	–0.04358(2)	0.51939(2)	0.0199(2)	0.0253(2)	0.0207(2)	0.0049(1)	0.0037(1)	0.0121(1)
N(1)	2*i*	0.2035(7)	0.3976(4)	0.9173(3)	0.036(3)	0.020(3)	0.016(2)	0.001(2)	0.005(2)	0.007(2)
N(2)	2*i*	0.171(1)	0.4382(4)	1.0678(4)	0.088(5)	0.013(3)	0.018(3)	–0.006(3)	0.015(3)	–0.001(2)
N(3)	2*i*	0.3253(9)	0.7553(4)	1.1508(5)	0.046(4)	0.023(3)	0.052(4)	0.006(3)	0.009(3)	0.007(3)
N(6)	2*i*	0.6818(7)	0.4430(4)	0.5505(4)	0.029(3)	0.024(3)	0.032(3)	0.005(2)	0.002(2)	0.014(2)
N(7)	2*i*	0.489(1)	0.3261(5)	0.4189(5)	0.065(5)	0.033(3)	0.041(4)	–0.006(3)	–0.010(3)	0.020(3)
N(8)	2*i*	0.318(1)	0.0576(5)	–0.0184(4)	0.069(5)	0.042(4)	0.021(3)	–0.014(3)	–0.006(3)	0.022(3)
N(9)	2*i*	0.2329(7)	–0.0319(4)	0.0616(3)	0.035(3)	0.029(3)	0.013(2)	0.001(2)	0.001(2)	0.015(2)
N(10)	2*i*	0.0216(8)	–0.1840(5)	–0.2777(4)	0.042(3)	0.050(4)	0.017(3)	0.008(3)	0.001(2)	0.019(3)
N(11)	2*i*	0.718(2)	0.4400(8)	0.2425(6)	0.14(1)	0.072(6)	0.052(5)	0.018(6)	0.026(6)	0.040(5)
O(1)	2*i*	0.1953(6)	0.0599(3)	0.6927(3)	0.032(2)	0.031(2)	0.012(2)	0.016(2)	0.005(2)	0.009(2)
O(2)	2*i*	0.0574(5)	0.0938(3)	0.5886(2)	0.012(2)	0.018(2)	0.009(2)	0.002(2)	0.000(1)	0.004(2)
O(3)	2*i*	0.0047(6)	–0.1595(4)	0.5569(3)	0.029(2)	0.040(3)	0.024(2)	–0.005(2)	0.001(2)	0.024(2)
O(4)	2*i*	0.2702(5)	0.0425(4)	0.4141(3)	0.018(2)	0.048(3)	0.026(2)	0.008(2)	0.005(2)	0.030(2)
O(5)	2*i*	0.4533(5)	–0.0823(3)	0.6147(3)	0.014(2)	0.027(2)	0.024(2)	0.005(2)	0.000(2)	0.019(2)
O(7)	2*i*	0.5129(5)	0.1030(3)	0.5868(3)	0.018(2)	0.015(2)	0.015(2)	–0.001(2)	0.004(2)	0.006(2)
O(8)	2*i*	0.2586(5)	–0.2238(3)	0.3997(3)	0.016(2)	0.014(2)	0.033(2)	–0.001(2)	0.008(2)	0.007(2)
O(12)	2*i*	0.1189(8)	0.3308(3)	1.0106(3)	0.085(4)	0.017(2)	0.012(2)	–0.010(2)	0.013(2)	0.002(2)
O(14)	2*i*	0.4554(7)	0.3079(4)	0.4968(4)	0.047(3)	0.030(3)	0.042(3)	–0.008(2)	–0.004(2)	0.020(2)
O(19)	2*i*	0.3973(8)	0.1136(4)	0.0785(3)	0.066(4)	0.037(3)	0.023(2)	–0.016(3)	–0.004(2)	0.022(2)

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