Jin-Long Yan*

Crystal structure of diethanol- $\kappa^1 O$ -bis(μ_2 - N -((2-oxidonaphthalen-1-yl)methylene)pyrazine-2-carbohydrazonato- $\kappa^5 N, O, O':O':N'$)-bis(nitrato- $\kappa^2 O, O'$)dieuropium(III), $C_{36}H_{32}N_{10}O_{12}Eu_2$

Table 1: Data collection and handling.

Crystal:	Plate, red
Size:	0.10 × 0.06 × 0.02 mm
Wavelength:	Cu $K\alpha$ radiation (1.54178 Å)
μ :	23.45 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω -scans
$\theta_{\max}$, completeness:	74.8°, >98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	18243, 3918, 0.094
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3025
$N(\text{param})_{\text{refined}}$:	273
Programs:	Bruker programs [1], SHELX [2]

<https://doi.org/10.1515/ncrs-2019-0772>

Received October 17, 2019; accepted December 31, 2019; available online January 21, 2020

Abstract

$C_{36}H_{32}N_{10}O_{12}Eu_2$, monoclinic, $C2/c$ (no. 15), $a = 16.445(2)$ Å, $b = 10.0920(14)$ Å, $c = 23.865(3)$ Å, $\beta = 100.115(8)^\circ$, $V = 3899.3(9)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0687$, $wR_{\text{ref}}(F^2) = 0.2065$, $T = 150(2)$ K.

CCDC no.: 1974929

The dinuclear title complex is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

The hydrazone ligand, N -((2-hydroxynaphthalen-1-yl)methylene)pyrazine-2-carbohydrazonic acid (H_2L) was prepared via the condensation of 2-pyrazinoylhydrazide and 2-hydroxy-1-naphthaldehyde according to the literature [4]. The reaction of europium(II) nitrate in ethanol under basic conditions yielded the title compound. The crystals of the title complex were obtained by slow evaporation of the ethanol solution.

Table 2: Fractional atomic coordinates and isotropic or equivalent displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Eu1	0.51082(3)	0.67657(6)	0.46830(2)	0.0254(2)
O1	0.5489(4)	0.4530(7)	0.4663(3)	0.0288(15)
O2	0.5135(5)	0.8478(7)	0.4134(3)	0.0314(17)
O3	0.6558(5)	0.6695(9)	0.5172(4)	0.050(2)
O4	0.5763(5)	0.8026(9)	0.5529(4)	0.044(2)
O5	0.7049(6)	0.7785(13)	0.5941(5)	0.073(3)
O6	0.3891(4)	0.5946(8)	0.4056(3)	0.0324(17)
H6	0.3435	0.5827	0.4161	0.049*
N1	0.6057(5)	0.2080(9)	0.4863(4)	0.0283(19)
N2	0.7337(5)	0.1093(9)	0.4364(4)	0.032(2)
N3	0.6309(5)	0.4872(10)	0.3987(4)	0.032(2)
N4	0.5948(5)	0.6144(9)	0.3949(4)	0.0291(19)
N5	0.6476(5)	0.7522(10)	0.5564(4)	0.036(2)
C1	0.6341(7)	0.0872(11)	0.4970(5)	0.034(2)
H1	0.6105	0.0332	0.5225	0.041*
C2	0.6978(6)	0.0362(12)	0.4722(5)	0.035(2)
H2	0.7162	−0.0519	0.4807	0.042*
C3	0.7035(6)	0.2300(11)	0.4240(4)	0.027(2)
H3	0.7263	0.2828	0.3976	0.032*
C4	0.6391(6)	0.2813(10)	0.4486(4)	0.026(2)
C5	0.6054(6)	0.4177(11)	0.4363(4)	0.028(2)
C6	0.6192(6)	0.6886(10)	0.3561(4)	0.026(2)
H6A	0.6551	0.6475	0.3342	0.032*
C7	0.5988(6)	0.8226(11)	0.3427(4)	0.028(2)
C8	0.5473(6)	0.8989(11)	0.3725(5)	0.030(2)
C9	0.5308(6)	1.0341(11)	0.3577(5)	0.032(2)
H9	0.4954	1.0834	0.3774	0.039*
C10	0.5640(7)	1.0947(11)	0.3163(5)	0.033(2)
H10	0.5532	1.1859	0.3086	0.040*
C11	0.6151(6)	1.0230(11)	0.2841(4)	0.028(2)

*Corresponding author: Jin-Long Yan, School of Science and Technology, Jiaozuo Teachers College, Jiaozuo 454001, P.R. China, e-mail: jinlong_yan@163.com. <https://orcid.org/0000-0003-2396-1499>

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
C12	0.6465(8)	1.0820(13)	0.2390(5)	0.044(3)
H12	0.6347	1.1729	0.2308	0.052*
C13	0.6927(7)	1.0157(13)	0.2067(5)	0.040(3)
H13	0.7107	1.0574	0.1754	0.048*
C14	0.7130(8)	0.8859(14)	0.2205(5)	0.045(3)
H14	0.7479	0.8392	0.1996	0.055*
C15	0.6834(8)	0.8219(12)	0.2645(5)	0.043(3)
H15	0.6976	0.7317	0.2724	0.052*
C16	0.6334(6)	0.8870(12)	0.2972(5)	0.033(2)
C17	0.3786(8)	0.5830(14)	0.3446(5)	0.046(3)
H17A	0.4099	0.6545	0.3296	0.056*
H17B	0.3194	0.5948	0.3281	0.056*
C18	0.4071(10)	0.4527(16)	0.3263(6)	0.063(4)
H18A	0.4009	0.4508	0.2847	0.095*
H18B	0.3738	0.3818	0.3389	0.095*
H18C	0.4653	0.4397	0.3431	0.095*

Experimental details

The structure was solved by direct methods and refined with the SHELX crystallographic software package [2]. The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters.

Discussion

Many hydrazones and their complexes have been found to show promising biological activities [3]. Particularly, the dysprosium(III) complexes of hydrazones derived from 2-pyrazinoylhydrazide and substituted salicylaldehyde show distinct single-molecule-magnet behaviour [4, 5]. In this work, the europium(III) complex of a pyrazinoylhydrazone

ligand was synthesized and characterized by X-ray diffraction.

As shown in the figure, there exists one discrete dimeric Eu(II) complex located around a center of inversion with the Eu-Eu distance of 3.9123(5) Å in the title crystal structure. Each europium(III) ion is eight-coordinated to one O₂N donor set from one L²⁻, one N atom and one μ²-O atom from the adjacent L²⁻ ligand (Symmetry code: (i) $-x + 1, -y + 1, -z + 1$), one O atom from one coordinated ethanol and two O atoms from one nitrate anion showing a bidentated coordination mode. These coordinations result in a distorted bicapped triangular prism coordination geometry. In the solid, pairs of intermolecular O—H...N hydrogen bonds between ethanol and terminal pyrazine N atom link dimers into an one-dimensional chain.

References

1. Bruker. SMART and SAINT. Bruker AXS Inc., Madison, WI, (USA) 2007.
2. Sheldrick, G. M.: Crystal structure refinement with SHELX. *Acta Crystallogr.* **C71** (2015) 3–8.
3. Ye, X.-P.; Wang, G.-J.; Pan, P.; Zhang, Z.-P.; Wu, W.-N.; Wang, Y.: Syntheses, Crystal structures and biological activities of two Cu(II) complexes with an acylhydrazone ligand bearing pyrrole unit. *Chin. J. Inorg. Chem.* **30** (2014) 2789–2795.
4. Tian, H.-Q.; Zhao, L.; Guo, Y.-N.; Tang, J.-K.; Liu, Z.-L.: Quadruple-CO₃²⁻-bridged octanuclear dysprosium(III) compound showing single-molecule magnet behaviour. *Chem. Commun.* **48** (2012) 708–710.
5. Tian, H.-Q.; Guo, Y.-N.; Zhao, L.; Tang, J.-K.; Liu, Z.-L.: Hexanuclear dysprosium(III) compound incorporating vertex- and edge-sharing Dy₃triangles exhibiting single-molecule-magnet behavior. *Inorg. Chem.* **50** (2011) 8688–8690.