He Yaling, Zeng Shilin, Zhang Peiling* and Xu Zhouqing

Crystal structure of diaqua-dinitrato- $\kappa^2 O, O'$ ((*Z*)-*N*-((*E*)-1-(pyrazin-2-yl)ethylidene)nicotinohydrazonato- $\kappa^3 N, N', O$)europium(II), $C_{12}H_{14}N_7O_9Eu$

Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.3 × 0.2 × 0.2 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	3.43 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART APEX, ω -scans
θ_{max} , completeness:	25°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	9021, 3265, 0.019
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 3023
$N(param)_{refined}$:	265
Programs:	Bruker programs [1], SHELX [2]

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

Atom	x	y	z	U_{iso}^*/U_{eq}
Eu1	0.707812(7)	0.561905(16)	0.307457(8)	0.02413(6)
C1	0.56651(17)	0.3739(4)	0.41599(19)	0.0365(6)
H1	0.5307	0.3713	0.3550	0.044*
C2	0.53898(18)	0.3045(4)	0.4854(2)	0.0407(7)
H2	0.4853	0.2573	0.4699	0.049*
C3	0.66294(19)	0.3749(4)	0.59151(19)	0.0372(6)
H3	0.6981	0.3774	0.6527	0.045*
C4	0.69197(18)	0.4456(3)	0.52348(19)	0.0295(6)
C5	0.77634(18)	0.5215(4)	0.54458(19)	0.0318(6)
C6	0.8338(2)	0.5324(5)	0.6427(2)	0.0533(9)
H6A	0.8869	0.5799	0.6432	0.080*
H6B	0.8423	0.4187	0.6696	0.080*
H6C	0.8091	0.6058	0.6781	0.080*
C7	0.89398(16)	0.6782(4)	0.41603(19)	0.0335(6)
C8	0.97780(17)	0.7587(4)	0.4292(2)	0.0353(6)
C9	0.99715(19)	0.8365(5)	0.3566(2)	0.0504(8)
H9	0.9577	0.8384	0.2976	0.060*
C10	1.0751(2)	0.9117(5)	0.3717(3)	0.0562(9)
H10	1.0884	0.9669	0.3236	0.067*
C11	1.13279(19)	0.9031(4)	0.4595(2)	0.0469(8)
H11	1.1853	0.9537	0.4699	0.056*
C12	1.04039(17)	0.7557(4)	0.5149(2)	0.0386(7)
H12	1.0286	0.7021	0.5643	0.046*
N1	0.64234(14)	0.4442(3)	0.43337(15)	0.0299(5)
N2	0.58747(15)	0.3036(3)	0.57386(16)	0.0410(6)
N3	0.79739(14)	0.5740(3)	0.47362(15)	0.0296(5)
N4	0.87724(14)	0.6442(3)	0.49366(16)	0.0365(5)
N5	1.11609(14)	0.8249(3)	0.53036(18)	0.0426(6)
N6	0.63022(15)	0.8962(3)	0.32196(16)	0.0350(5)
N7	0.68730(16)	0.6104(3)	0.11136(16)	0.0393(6)
O1	0.84578(11)	0.6475(3)	0.33349(13)	0.0390(5)
O2	0.70859(12)	0.8844(3)	0.34105(15)	0.0415(5)
O3	0.58882(12)	0.7567(2)	0.30913(14)	0.0385(5)
O4	0.59448(16)	1.0371(3)	0.31490(19)	0.0560(6)

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Abstract

$C_{12}H_{14}N_7O_9Eu$, monoclinic, $P2_1/c$ (no. 14), $a = 16.770(3)$ Å, $b = 7.7185(16)$ Å, $c = 15.165(3)$ Å, $\beta = 108.409(3)^\circ$, $V = 1862.5(7)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0174$, $wR_{ref}(F^2) = 0.0453$, $T = 296(2)$ K.

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The crystal structure is shown in the figure. Hydrogen atoms are omitted for clarity. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

All chemicals were commercially purchased except for (*E*)-*N*'-(1-(pyrazin-2-yl)ethylidene)nicotinohydrazide (HL) which

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Table 2 (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
O5	0.66480(14)	0.7272(3)	0.15723(14)	0.0454(5)
O6	0.73079(13)	0.4877(3)	0.15779(14)	0.0401(5)
O7	0.66750(19)	0.6168(4)	0.02697(16)	0.0711(8)
O8	0.57973(12)	0.4252(2)	0.21120(14)	0.0359(5)
H8A	0.5410	0.4402	0.2355	0.054*
H8B	0.5884	0.3174	0.2074	0.054*
O9	0.75003(12)	0.2650(2)	0.32711(13)	0.0372(4)
H9A	0.7103	0.2038	0.3342	0.056*
H9B	0.7931	0.2555	0.3749	0.056*

was synthesized according to the literature [3]. A mixture of HL (0.05 mmol, 12.1 mg) and Eu(NO₃)₃·6H₂O (0.10 mmol, 44.6 mg) dissolved in ethanol (3 mL) and H₂O (3 mL) was placed in a glass bottle (10 mL) at room temperature. Pale yellow block crystals suitable for single-crystal X-ray diffraction were obtained after 3 days.

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

Hydrazones are a class of compounds which are well known for their antimicrobial activity [4, 5]. But as hydrazones are prone to undergo degradation and bacteria can develop resistance to them, complexation of hydrazones with bio-compatible metal ions is useful in this regard for long-term effectiveness [6]. In the course of our studies on the chemistry of lanthanide compounds, we have prepared and characterized a number of chelates containing hydrazones as ligands. Herein we report a new Eu(III) complexes derived from

(*E*)-*N'*-(1-(pyrazin-2-yl)ethylidene)nicotinohydrazide (HL), producing complexes of the type [Eu(L)(NO₃)₂(H₂O)₂].

As reported, the title complex is a mononuclear coordinational compound in which the nine coordinated Eu³⁺ is surrounded by one hydrozonate, two nitrate ligands and two water molecules. The bond lengths of Eu—O and Eu—N (2.317(19)–2.646(2) Å) in the title complex are comparable with those found in our previous work [7]. In the solid state, the three dimension supermolecules are constructed by inter-molecular O—H···O and O—H···N hydrogen bonds between the coordinated water molecules and the N atoms of ligand and the O atoms of the nitrate anions.

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