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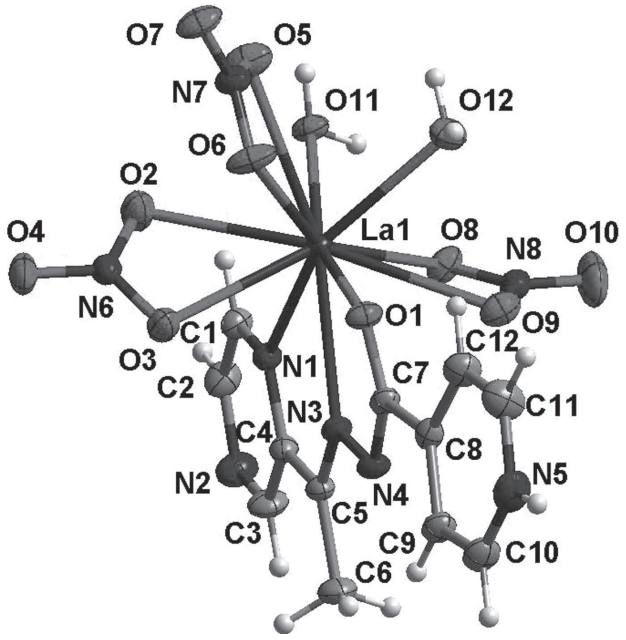
Crystal structure of diaqua-(*N*-(1-(pyrazin-2-yl)ethylidene)pyridin-1-ium-4-carbohydrazonate-κ^3*N*,*N*′,*O*)-tris[nitrato-κ^2*O*,*O*′)lanthanum(III), $C_{12}H_{15}N_8O_{12}La$



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Abstract

$C_{12}H_{15}N_8O_{12}La$, monoclinic, $P2_1/c$ (no. 14), $a = 8.3267(7)$ Å, $b = 28.348(2)$ Å, $c = 9.1097(8)$ Å, $\beta = 107.5410(10)°$, $V = 2050.3(3)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0236$, $wR_{ref}(F^2) = 0.0559$, $T = 296(2)$ K.

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

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Table 1: Data collection and handling.

Crystal:	Yellow block
Size:	0.30 × 0.20 × 0.20 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	2.16 mm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX, ω
θ_{max}, completeness:	25.0°, 97%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10367, 3601, 0.031
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3194
$N(param)_{refined}$:	305
Programs:	SHELX [1], Bruker [2]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
La1	0.86828(2)	0.15301(2)	0.79298(2)	0.02360(7)
C1	1.2067(4)	0.21208(12)	1.0869(4)	0.0373(8)
H1	1.1920	0.2332	1.0058	0.045*
C2	1.3175(4)	0.22363(13)	1.2289(4)	0.0438(9)
H2	1.3731	0.2525	1.2410	0.053*
C3	1.2594(5)	0.15434(13)	1.3231(4)	0.0449(9)
H3	1.2761	0.1333	1.4046	0.054*
C4	1.1461(4)	0.14220(12)	1.1830(3)	0.0314(7)
C5	1.0495(4)	0.09800(11)	1.1610(3)	0.0302(7)
C6	1.0770(5)	0.06339(13)	1.2909(4)	0.0449(9)
H6A	1.1955	0.0581	1.3360	0.067*
H6B	1.0224	0.0341	1.2523	0.067*
H6C	1.0307	0.0758	1.3674	0.067*
C7	0.7456(4)	0.04803(10)	0.8674(4)	0.0298(7)
C8	0.6317(4)	0.00601(11)	0.8394(4)	0.0322(7)
C9	0.6575(4)	−0.03089(11)	0.9439(4)	0.0392(8)
H9	0.7484	−0.0303	1.0333	0.047*
C10	0.5485(5)	−0.06815(12)	0.9147(5)	0.0466(9)
H10	0.5642	−0.0928	0.9850	0.056*
C11	0.3920(5)	−0.03459(13)	0.6817(5)	0.0560(10)
H11	0.3013	−0.0366	0.5924	0.067*
C12	0.4968(5)	0.00377(12)	0.7069(4)	0.0456(9)
H12	0.4772	0.0281	0.6352	0.055*
N1	1.1206(3)	0.17200(9)	1.0620(3)	0.0313(6)
N2	1.3462(4)	0.19471(11)	1.3475(3)	0.0494(8)
N3	0.9415(3)	0.09231(8)	1.0270(3)	0.0296(6)
N4	0.8467(3)	0.05117(9)	1.0083(3)	0.0327(6)
N5	0.4214(4)	−0.06923(12)	0.7871(4)	0.0510(9)
N6	1.1969(3)	0.12365(11)	0.7267(3)	0.0392(7)
N7	0.8166(4)	0.15276(9)	0.4318(3)	0.0352(6)
N8	0.6465(4)	0.18546(11)	0.9964(3)	0.0389(7)
O1	0.7315(3)	0.07597(7)	0.7553(2)	0.0357(5)
O2	1.1533(3)	0.16562(10)	0.7161(3)	0.0565(7)

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O3	1.1149(3)	0.09569(9)	0.7833(3)	0.0540(7)
O4	1.3147(3)	0.10959(10)	0.6829(3)	0.0547(7)
O5	0.8429(4)	0.19200(8)	0.4934(3)	0.0562(7)
O6	0.8133(4)	0.11807(9)	0.5121(3)	0.0545(7)
O7	0.7957(4)	0.14813(8)	0.2917(3)	0.0517(7)
O8	0.7794(3)	0.20594(8)	0.9930(3)	0.0445(6)
O9	0.6243(3)	0.14498(8)	0.9423(3)	0.0479(6)
O10	0.5469(4)	0.20499(13)	1.0501(4)	0.0851(11)
O11	0.9116(3)	0.24303(7)	0.7732(2)	0.0389(5)
H11A	0.8717	0.2517	0.6795	0.058*
H11B	0.8852	0.2601	0.8583	0.058*
O12	0.5670(3)	0.17786(10)	0.6498(3)	0.0494(6)
H12A	0.5137	0.1552	0.5911	0.074*
H12B	0.5685	0.2022	0.5927	0.074*
H5	0.353(6)	−0.0913(17)	0.763(5)	0.079(16)*

Source of material

All chemicals were commercially purchased except for (*E*)-*N*′-(1-(pyrazin-2-yl)ethylidene)isonicotinohydrazide (*HL*) which was synthesized according to the literature [3]. A mixture of *HL* (0.05 mmol, 12.1 mg) and $La(NO_3)_3 \cdot 6\ H_2O$ (0.10 mmol, 43.3 mg) disolved in a absolute methanol (3 mL) and H_2O (3 mL) was placed in a glass bottle (10 mL) at room temperature. Green block crystal were obtained after 3 days.

Experimental details

The structure was solved by Direct Methods and refined with the SHELX crystallographic software package [1, 2]. The hydrogen atoms were placed at calculated positions and refined as riding atoms with fixed isotropic displacement parameters.

Comment

Hydrazones are a class of compounds which are well known for their antimicrobial activity [4]. But as hydrazones are prone to undergo degradation and bacteria can develop resistance to them, complexation of hydrazones with biocompatible metal ions is useful in this regard for long-term effectiveness [5]. 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 La(III) complex derived from (*E*)-*N*′-(1-(pyrazin-2-yl)ethylidene)isonicotinohydrazide, producing the title complex.

The title complex is a mononuclear coordination compound in which the eleven-coordinated La^{3+} is surrounded by three nitrates, two water molecules and one independent $\kappa^3 N,N';O$ coordinated organic ligand thus giving distorted monocapped pentagonal prism geometry. The bond lengths of La—O and La—N (2.439(8)—2.893(6) Å) in the title complex are comparable with those found in our previous work [6]. In the solid state, the three dimensional connection is constructed by the O—H···O and O—H···N hydrogen bonds.

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