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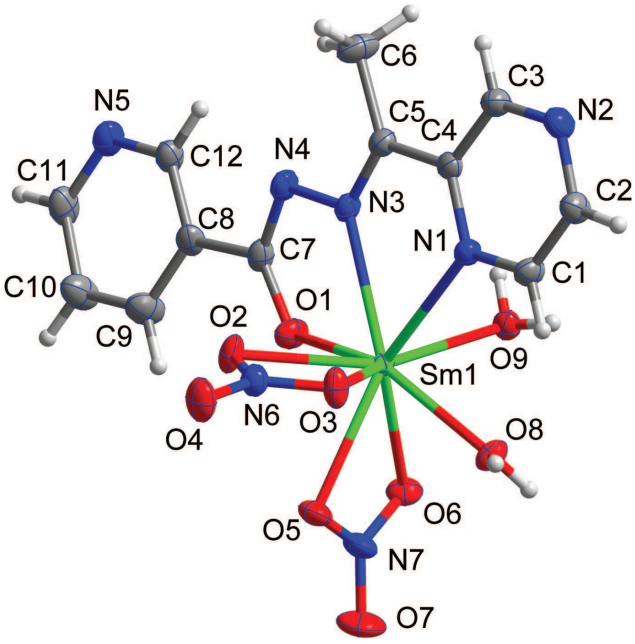
Crystal structure of diaqua-(*N*-(1-(pyrazin-2-yl) ethylidene)nicotinohydrazonato-κ^3*N*,*N*′,*O*)-bis (nitrato-κ^2*O*,*O*′)samarium(III), $C_{12}H_{14}N_7O_9Sm$



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Abstract

$C_{12}H_{14}N_7O_9Sm$, monoclinic, $P2_1/c$ (no. 14), $a = 16.770(3)$ Å, $b = 7.7185(16)$ Å, $c = 15.165(3)$ Å, $\beta = 108.409(3)°$, $V = 1862.5(7)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0262$, $wR_{ref}(F^2) = 0.0672$, $T = 296(2)$ 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:	3.22 mm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX CCD, ω
θ_{max}, completeness:	25.0°, 97%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9083, 3264, 0.035
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2972
$N(param)_{refined}$:	264
Programs:	SHELX [2], Bruker [3]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Sm1	0.70738(2)	0.55866(2)	0.30975(2)	0.02184(9)
C1	0.5651(2)	0.3708(5)	0.4174(3)	0.0385(9)
H1	0.5294	0.3677	0.3564	0.046*
C2	0.5370(3)	0.3013(5)	0.4869(3)	0.0421(10)
H2	0.4835	0.2527	0.4712	0.050*
C3	0.6616(3)	0.3759(5)	0.5939(3)	0.0401(9)
H3	0.6967	0.3798	0.6552	0.048*
C4	0.6906(2)	0.4458(4)	0.5249(3)	0.0305(9)
C5	0.7751(2)	0.5216(5)	0.5456(3)	0.0331(9)
C6	0.8325(3)	0.5340(7)	0.6441(3)	0.0560(13)
H6A	0.8887	0.5573	0.6442	0.084*
H6B	0.8314	0.4266	0.6756	0.084*
H6C	0.8138	0.6261	0.6754	0.084*
C7	0.8932(2)	0.6748(5)	0.4164(3)	0.0342(9)
C8	0.9779(2)	0.7560(5)	0.4288(3)	0.0366(9)
C9	0.9969(3)	0.8315(6)	0.3552(3)	0.0548(12)
H9	0.9574	0.8310	0.2961	0.066*
C10	1.0748(3)	0.9079(6)	0.3694(4)	0.0592(13)
H10	1.0878	0.9621	0.3209	0.071*
C11	1.1327(3)	0.9013(6)	0.4576(4)	0.0496(12)
H11	1.1852	0.9512	0.4675	0.060*
C12	1.0400(2)	0.7564(5)	0.5143(3)	0.0388(9)
H12	1.0281	0.7053	0.5643	0.047*
N1	0.6411(2)	0.4416(3)	0.4348(2)	0.0309(7)
N2	0.5850(2)	0.3028(4)	0.5756(2)	0.0432(8)
N3	0.7962(2)	0.5732(4)	0.4746(2)	0.0321(7)
N4	0.87633(19)	0.6442(4)	0.4942(2)	0.0374(8)
N5	1.1159(2)	0.8258(4)	0.5294(3)	0.0451(9)
N6	0.6314(2)	0.8925(4)	0.3243(2)	0.0361(7)
N7	0.6878(2)	0.6083(5)	0.1146(2)	0.0434(5)
O1	0.84449(16)	0.6429(4)	0.33325(19)	0.0415(7)

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O2	0.71000(17)	0.8799(3)	0.3434(2)	0.0423(7)
O3	0.58929(16)	0.7532(3)	0.3117(2)	0.0398(7)
O4	0.5960(2)	1.0339(3)	0.3166(3)	0.0557(9)
O5	0.66502(18)	0.7248(3)	0.16133(18)	0.0434(5)
O6	0.73047(18)	0.4833(4)	0.1611(2)	0.0399(6)
O7	0.6689(2)	0.6150(5)	0.0303(2)	0.0714(10)
O8	0.57986(17)	0.4242(3)	0.2155(2)	0.0375(7)
H8B	0.5725	0.3481	0.1732	0.056*
H8A	0.5309	0.4560	0.2141	0.056*
O9	0.74889(17)	0.2634(3)	0.32872(19)	0.0375(6)
H9A	0.7080	0.2013	0.3317	0.056*
H9B	0.7897	0.2527	0.3787	0.056*

Source of material

All chemicals were commercially purchased except for (*E*)—*N*′-(1-(pyrazin-2-yl)ethylidene)nicotinohydrazide (*HL*) which was synthesized according to the literature [1]. A mixture of *HL* (0.05 mmol, 12.1 mg) and $Sm(NO_3)_3{\cdot}6\ H_2O$ (0.10 mmol, 44.7 mg) disolved in absolute ethanol (3 mL) and H_2O (3 mL) was placed in a glassbottle (10 mL) at room temperature. Pale yellow block crystals were obtained after a few days.

Experimental details

The structure was solved by Direct Methods and refined with the SHELX crystallographic software package [2, 3]. The hydrogen atoms were placed at calculated positions and refined as riding atoms with isotropic displacement parameters.

Comment

Some hydrazones are well known for their antimicrobial activity [1, 4]. But as hydrazones are prone to undergo degradation and bacteria can develop resistance to them, complexation of hydrazones with biocompatible metal ions may be useful for long-term effectiveness [5]. In the course of our studies on the chemistry of lanthanide compounds, we have prepared and characterized hydrazones as ligands.

As depicted in the figure, the title complex is a mononuclear coordinational compound in which the six-coordinated Sm^{3+} is surrounded by one acylhydrazone ligand, two water molecules an two nitrato ligands, thus giving distorted octahedral geometry. The bond lengths of Sm—O and Sm—N (2.307(2)—2.641(3) Å) in the title complex are comparable with those found in our previous work [6]. In the solid state, the three dimensional framework is constructed by the intermolecular O—H···O and O—H···N hydrogen bonds between all moieties.

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