Crystal structure of triaquabis(vanillin-*O*,*O*′)nitritogadolinium(III), $Gd(H_2O)_3(C_8H_7O_3)_2(NO_2)$

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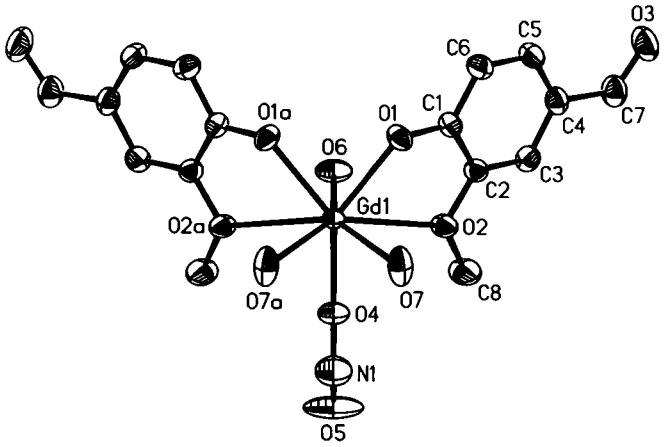

Abstract

$C_{16}H_{20}GdNO_{11}$, orthorhombic, *Pnma* (no. 62), $a = 7.784(2)$ Å, $b = 21.977(4)$ Å, $c = 11.129(2)$ Å, $V = 1903.9$ Å^3, $Z = 4$, $R_{gt}(F) = 0.018$, $wR_{ref}(F^2) = 0.048$, $T = 293$ K.

Source of material

Adamantaneamine (0.30 g, 2.0 mmol) and vanillin (0.31 g, 2.2 mmol) in absolute ethanol (30 mL) were stirred and refluxed for 4 h, producing a light yellow solution, to which a solution of $Gd(NO_3)_3 \cdot 6H_2O$ (0.45 g, 1.0 mmol) in 10 mL absolute ethanol was added with stirring. The mixture was then further stirred at room temperature for 5 h. Slow evaporation afforded the title complex as crystals suitable for the X-ray structure determination.

Discussion

The title complex crystallizes isostructurally with triaquabis(vanillin-*O*,*O*′)nitritoerbium(III) reported by us [1]. The crystal structure is built up by triaquabis(vanillin-*O*,*O*′)nitritogadolinium(III) complex molecules, within which the Gd atoms are each surrounded by four O atoms from two vanillin ligands, three aqua molecules and one O atom of nitrito ligand, to which nitrato group was reduced by adamantaneimine. The environment around Gd atom can be described as a distorted square antiprism with O1, O7, O7a, O1a (plane I) and O2, O4, O2a, O6 (plane II) (symmetry code: (a) $x,-y+½,z$) at the tetragonal bases. The dihedral angle between their mean planes is 3.86(8)°. The distances between Gd and the two planes are –1.095(1) Å and 1.362(1) Å, respectively. The complex molecules are connected to each other through strong intramolecular hydrogen bonds with d(O6···O5b) = 2.664(2) Å and ∠O6–H···O5b = 172.2°, d(O6···O5c) = 2.673(2) Å and ∠O6–H···O5c = 178.1°, d(O7···O1d) = 2.678(2) Å and ∠O7–H···O1d = 168.3°, d(O7···O3e) = 2.821(2) Å and ∠O7–H···O3e = 171.4° (symmetry codes: (b) $x-1,y,z$; (c) $x-½,y,-z+½$; (d) $x+½,y,-z+3/2$; (e) $-x,-y+1,-z+1$) to form the three-dimensional structure.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.12 × 0.20 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	35.45 cm^{-1}
Diffractometer, scan mode:	Rigaku R-AXIS RAPID, ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	17407, 2236
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2158
$N(param)_{refined}$:	139
Programs:	SHELXS-97 [2], SHELXL-97 [3]

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

Atom	Site	x	y	z	U_{iso}
H(61)	4*c*	–0.0950	¼	0.3501	0.036
H(62)	4*c*	0.0707	¼	0.2937	0.036
H(71)	8*d*	0.3502	0.3128	0.7512	0.045
H(72)	8*d*	0.3131	0.3546	0.6698	0.045
H(3)	8*d*	0.0756	0.4571	0.3309	0.029
H(5)	8*d*	–0.3933	0.4647	0.4978	0.035
H(6)	8*d*	–0.3293	0.3765	0.6048	0.035
H(7)	8*d*	–0.1116	0.5408	0.2870	0.038
H(8A)	8*d*	0.2467	0.3780	0.2794	0.060
H(8B)	8*d*	0.4045	0.3525	0.3544	0.060
H(8C)	8*d*	0.3424	0.4192	0.3756	0.060

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}
Gd(1)	4*c*	0.17594(2)	¼	0.54402(1)	0.01487(8)	0.01818(8)	0.01557(8)	0	0.00083(5)	0
O(1)	8*d*	–0.0444(2)	0.31495(7)	0.5960(1)	0.0270(8)	0.0236(7)	0.0261(8)	0.0070(6)	0.0087(6)	0.0057(6)
O(2)	8*d*	0.1865(2)	0.35830(8)	0.4465(1)	0.0206(8)	0.0277(9)	0.0270(8)	0.0027(6)	0.0041(6)	0.0083(6)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(3)	8*d*	–0.3294(3)	0.55702(9)	0.3522(2)	0.045(1)	0.0282(9)	0.051(1)	0.0108(8)	–0.0096(9)	0.0050(8)
O(4)	4*c*	0.4286(3)	¼	0.4396(2)	0.019(1)	0.037(1)	0.037(1)	0	0.007(1)	0
O(5)	4*c*	0.6823(3)	¼	0.3534(3)	0.019(1)	0.140(4)	0.023(1)	0	0.004(1)	0
O(6)	4*c*	0.0244(3)	¼	0.3599(2)	0.020(1)	0.051(1)	0.020(1)	0	–0.0006(9)	0
O(7)	8*d*	0.3222(2)	0.31584(8)	0.6822(2)	0.059(1)	0.0217(8)	0.0312(9)	0.0018(8)	–0.0240(8)	–0.0034(7)
N(1)	4*c*	0.5900(5)	¼	0.4434(3)	0.035(2)	0.052(2)	0.036(2)	0	0.004(1)	0
C(1)	8*d*	–0.0858(3)	0.3640(1)	0.5354(2)	0.024(1)	0.021(1)	0.022(1)	0.0029(8)	0.0009(8)	–0.0013(8)
C(2)	8*d*	0.0321(3)	0.3892(1)	0.4521(2)	0.020(1)	0.022(1)	0.022(1)	0.0010(8)	–0.0017(8)	–0.0003(8)
C(3)	8*d*	–0.0063(3)	0.44089(9)	0.3878(2)	0.025(1)	0.024(1)	0.024(1)	–0.0018(9)	–0.0025(9)	0.0024(8)
C(4)	8*d*	–0.1657(3)	0.4697(1)	0.4042(2)	0.029(1)	0.020(1)	0.028(1)	0.0025(8)	–0.0078(9)	–0.0005(9)
C(5)	8*d*	–0.2838(3)	0.4451(1)	0.4858(2)	0.024(1)	0.025(1)	0.037(1)	0.0057(9)	–0.003(1)	–0.002(1)
C(6)	8*d*	–0.2458(3)	0.3933(1)	0.5497(2)	0.025(1)	0.028(1)	0.035(1)	0.004(1)	0.006(1)	0.0022(9)
C(7)	8*d*	–0.1988(3)	0.5264(1)	0.3420(2)	0.038(1)	0.025(1)	0.033(1)	0.002(1)	–0.008(1)	0.003(1)
C(8)	8*d*	0.3050(3)	0.3786(1)	0.3567(3)	0.030(1)	0.040(1)	0.050(2)	0.004(1)	0.016(1)	0.019(1)

References

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