Crystal structure of tris(nitrato-*O,O'*)bis(1,10-phenanthroline-*N,N'*)-cerium(III), $\text{Ce}(\text{NO}_3)_3(\text{C}_{12}\text{H}_8\text{N}_2)_2$

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Received August 17, 2003, accepted in revised form and available on-line November 16, 2003; CCDC-No. 1267/1139

Abstract

$\text{C}_{24}\text{H}_{16}\text{CeN}_7\text{O}_9$, monoclinic, $C12/c1$ (No. 15), $a = 11.162(2) \text{ \AA}$, $b = 18.070(4) \text{ \AA}$, $c = 13.102(3) \text{ \AA}$, $\beta = 100.56(3)^\circ$, $V = 2597.9 \text{ \AA}^3$, $Z = 4$, $R_{\text{gt}}(F) = 0.021$, $wR_{\text{ref}}(F^2) = 0.056$, $T = 293 \text{ K}$.

Source of material

To a solution of 1,10-phenanthroline (0.5 mmol) and lysine (0.5 mmol) in 15 mL of hot water was added $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (0.5 mmol). The mixture was stirred for 2 h, resulting in a light yellow solution. The yellow prism crystals suitable for structure determination were isolated by slow evaporation of the solvent at room temperature.

Experimental details

Lower symmetry space groups that break the 2-fold axis were tested. No indications of lower symmetry were observed. Thus, the final refinement was done using the space group $C2/c$. We have not clear explanations for the extremely high U_{ij} values for the positions O4, O5 and N4 atoms which were also observed in the complex $\text{Bi}(\text{NO}_3)_3(\text{phen})_2$ [1].

Discussion

Rare earth metal complexes are of growing importance because of their chemical [2], biochemical and medicinal applications [3]. A series of isostructural lanthanum-phen-nitrato complexes with general formula $\text{Ln}(\text{NO}_3)_3(\text{phen})_2$ ($\text{Ln} = \text{La}$ [4], Gd [5], Pr , Nd , Sm , Eu , Dy [6], Lu [7]) have been reported.

$\text{Ce}(\text{NO}_3)_3(\text{phen})_2$ is another member of the $\text{Ln}(\text{NO}_3)_3(\text{phen})_2$ family. The crystal structure is built up by two 1,10-phenanthroline molecules and three nitrato groups. The Ce atom is surrounded by six O atoms from three nitrato groups and four N atoms from two 1,10-phenanthroline ligands. The environment around Ce atom can be described as a distorted dicapped square antiprism with N1, N2, O4, O2a and N1a, N2a, O4a, O2 (symmetry code: (a) $1-x, y, 0.5-z$) at the tetragonal bases. The distances between Ce, capped O1 atom and the base plane are $-1.096(2) \text{ \AA}$ and $1.457(2) \text{ \AA}$, respectively. The average bond length of Ce—O and Ce—N are $2.566 \text{ \AA}$ and $2.641 \text{ \AA}$, respectively.

Table 1. Data collection and handling.

Crystal:	yellow prism, size $0.08 \times 0.20 \times 0.30 \text{ mm}$
Wavelength:	Mo $K\alpha$ radiation ($0.71073 \text{ \AA}$)
μ :	18.19 cm^{-1}
Diffractometer, scan mode:	Rigaku R-Axis RAPID, ω
$2\theta_{\text{max}}$:	54.92°
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$:	5799, 2967
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2822
$N(\text{param})_{\text{refined}}$:	187
Programs:	SHELXS-97 [8], SHELXL-97 [9]

Table 2. Atomic coordinates and displacement parameters (in $\text{\AA}^2$).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}
H(1)	8f	0.3395	0.1911	0.0881	0.061
H(2)	8f	0.2640	0.0705	0.0843	0.079
H(3)	8f	0.2535	0.0102	0.2378	0.082
H(5)	8f	0.2718	0.0166	0.4331	0.087
H(7)	8f	0.5036	0.3667	-0.0063	0.062
H(8)	8f	0.5376	0.3202	-0.1657	0.071
H(9)	8f	0.6089	0.2012	-0.1753	0.067
H(11)	8f	0.6828	0.0830	-0.0836	0.079

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Ce(1)	4e	1/2	0.327500(8)	1/4	0.0449(1)	0.02607(9)	0.02783(9)	0	0.00913(6)	0
O(1)	8f	0.6816(2)	0.3362(1)	0.4031(1)	0.062(1)	0.058(1)	0.0381(9)	−0.0035(8)	0.0052(8)	0.0003(7)
O(2)	8f	0.7193(2)	0.3711(1)	0.2549(1)	0.0546(9)	0.057(1)	0.0471(9)	−0.0049(8)	0.0141(7)	0.0022(8)
O(3)	8f	0.8601(2)	0.3765(2)	0.3914(2)	0.049(1)	0.114(2)	0.096(2)	−0.010(1)	−0.009(1)	−0.000(2)
O(4)	8f	0.4872(2)	0.4559(1)	0.1674(2)	0.084(2)	0.046(1)	0.103(2)	−0.005(1)	−0.003(1)	0.031(1)
O(5)	4e	1/2	0.5588(2)	1/4	0.122(4)	0.027(2)	0.298(8)	0	−0.035(4)	0
N(1)	8f	0.3675(2)	0.2044(1)	0.2405(1)	0.057(1)	0.0349(9)	0.0315(9)	−0.0077(8)	0.0096(8)	−0.0029(7)
N(2)	8f	0.5618(2)	0.2791(1)	0.0785(1)	0.055(1)	0.0395(9)	0.0292(8)	0.0077(8)	0.0100(7)	0.0040(7)
N(3)	8f	0.7571(2)	0.3613(1)	0.3513(2)	0.049(1)	0.043(1)	0.052(1)	0.0049(9)	0.0034(9)	−0.0055(9)
N(4)	4e	1/2	0.4918(2)	1/4	0.055(2)	0.029(2)	0.168(5)	0	−0.015(2)	0
C(1)	8f	0.3327(3)	0.1674(1)	0.1524(2)	0.075(2)	0.045(1)	0.035(1)	−0.014(1)	0.012(1)	−0.0088(9)
C(2)	8f	0.2892(3)	0.0953(2)	0.1495(2)	0.098(2)	0.050(2)	0.051(1)	−0.025(1)	0.018(1)	−0.020(1)
C(3)	8f	0.2811(3)	0.0606(2)	0.2389(2)	0.104(2)	0.039(1)	0.066(2)	−0.026(1)	0.028(2)	−0.013(1)
C(4)	8f	0.3127(3)	0.0982(1)	0.3337(2)	0.077(2)	0.037(1)	0.050(1)	−0.012(1)	0.021(1)	−0.001(1)
C(5)	8f	0.2997(4)	0.0667(2)	0.4310(2)	0.118(3)	0.042(1)	0.066(2)	−0.019(2)	0.036(2)	0.007(1)
C(6)	8f	0.3569(2)	0.1703(1)	0.3305(2)	0.051(1)	0.034(1)	0.036(1)	−0.0041(8)	0.0128(9)	−0.0010(8)
C(7)	8f	0.5359(3)	0.3176(1)	−0.0090(2)	0.070(2)	0.051(1)	0.036(1)	0.012(1)	0.013(1)	0.011(1)
C(8)	8f	0.5551(3)	0.2903(2)	−0.1043(2)	0.074(2)	0.075(2)	0.030(1)	0.011(1)	0.011(1)	0.012(1)
C(9)	8f	0.5979(3)	0.2214(2)	−0.1099(2)	0.066(2)	0.074(2)	0.030(1)	0.004(1)	0.014(1)	−0.005(1)
C(10)	8f	0.6264(2)	0.1786(1)	−0.0197(2)	0.056(1)	0.049(1)	0.037(1)	0.000(1)	0.014(1)	−0.0071(9)
C(11)	8f	0.6717(3)	0.1052(2)	−0.0194(2)	0.099(2)	0.054(2)	0.050(1)	0.012(2)	0.028(2)	−0.015(1)
C(12)	8f	0.6090(2)	0.2107(1)	0.0739(2)	0.046(1)	0.037(1)	0.031(1)	0.0017(8)	0.0110(8)	−0.0013(8)

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