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

Y.-L. Feng* and G.-L. Zhao

Zhejiang Normal University, Institute of Physical Chemistry, Jinhua, Zhejiang, 321004 P. R. China

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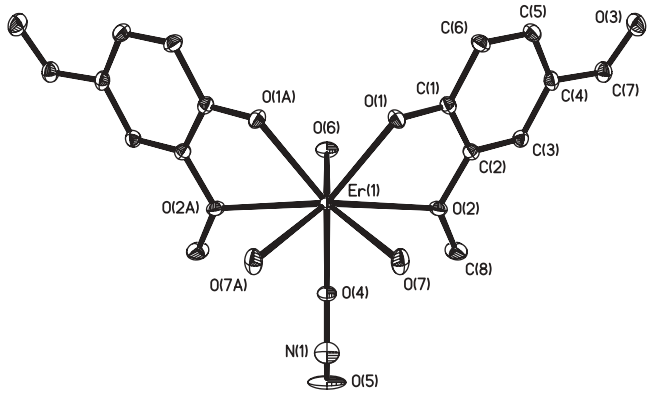

Abstract

$C_{16}H_{20}ErNO_{11}$, orthorhombic, *Pnma* (No. 62), a = 7.744(2) Å, b = 21.929(4) Å, c = 11.013(2) Å, V = 1870.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.020, $wR_{ref}(F^2)$ = 0.083, T = 293 K.

Source of material

Adamantaneamine (2.0 mmol) in 15.0 ml CH_3OH was added to a solution of $Er(NO_3)_3 \cdot 5H_2O$ (1.0 mmol) dissolved in 10.0 ml CH_3OH, producing a colorless solution, to which 2.2 mmol vanillin was added under continuous stirring. The mixture was then further stirred for 5 h. Slow evaporation afforded the title complex crystals for the X-ray structure determination.

Discussion

The crystal structure of the title compound is built up by triaquabis(vanillin-*O*,*O′*)nitritoerbium(III) complex molecules, within which the Er atoms are each surrounded by four O atoms from two vanillin ligands, three H_2O molecules and one O atom of nitrito ligand, to which nitrato group was reduced by adamantaneimine. The geometry around Er atom can be described as a distorted square antiprism with O1, O7, O7*a*, O1*a* (plane I) and O2, O4, O2*a*, O6 (plane II) (symmetry code: *a*: *x*, –*y*+1/2, *z*) at the tetragonal bases (dihedral angle between their mean planes is 3.3(1)°). The distances between Er and the two planes are –1.075(2) Å and 1.344(2) Å, respectively. The bond lengths of Er—O(phenato), Er—O(methyloxy) and Er—O(nitrito) are 2.260(3) Å, 2.585(3) Å and 2.236(4) Å, respectively. The average bond length of Er—O(water) is 2.330 Å. To form the three-dimensional structure, the complex molecules are connected to each orther through strong intramolecular hydrogen bonds with d(O6–H···O5*b*) = 2.677(5) Å and ∠O6–H···O5*b* = 172.2°, d(O6–H···O5*c*) = 2.677(6) Å and ∠O6–H···O5c = 179.6°, d(O7–H···O1*d*) = 2.688(4) Å and ∠O7–H···O1*d* = 167.8°, d(O7–H···O3e) = 2.839(5) Å and ∠O7–H···O3*e* = 170.6° (symmetry codes: *b*: *x*–1, *y*, *z*; *c*: *x*–1/2, *y*, –*z*+1/2; *d*: *x*+1/2, *y*, –*z*+3/2; *e*: –x, –y+1, –z+1, respectively).

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.12 × 0.20 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	45.50 cm^{-1}
Diffractometer, scan mode:	Rigaku R-AXIS RAPID, φ/ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3792, 2125
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1902
$N(param)_{refined}$:	139
Programs:	SHELXS-97 [1], SHELXL-97 [2]

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

Atom	Site	x	y	z	U_{iso}
H(61)	4*c*	–0.0958	1/4	0.3522	0.032
H(62)	4*c*	0.0698	1/4	0.2958	0.032
H(71)	8*d*	0.3470	0.3113	0.7481	0.039
H(72)	8*d*	0.3099	0.3531	0.6667	0.039
H(3)	8*d*	0.0760	0.4564	0.3305	0.027
H(5)	8*d*	–0.3939	0.4643	0.4987	0.031
H(6)	8*d*	–0.3295	0.3760	0.6060	0.032
H(7)	8*d*	–0.1108	0.5404	0.2874	0.035
H(8A)	8*d*	0.2492	0.3772	0.2778	0.054
H(8B)	8*d*	0.4070	0.3517	0.3528	0.054
H(8C)	8*d*	0.3449	0.4184	0.3740	0.054

* Correspondence author (e-mail: jhfyl@mail.jhptt.zj.cn)

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}
Er(1)	4*c*	0.17306(3)	1/4	0.54339(2)	0.0141(2)	0.0158(2)	0.0143(2)	0	0.00056(8)	0
O(1)	8*d*	–0.0436(4)	0.3136(1)	0.5968(3)	0.024(1)	0.020(1)	0.025(1)	0.006(1)	0.008(1)	0.006(1)
O(2)	8*d*	0.1876(3)	0.3571(2)	0.4460(3)	0.017(1)	0.024(2)	0.029(2)	0.003(1)	0.004(1)	0.007(1)
O(3)	8*d*	–0.3292(4)	0.5573(2)	0.3531(3)	0.041(2)	0.025(2)	0.047(2)	0.009(1)	–0.009(2)	0.006(2)
O(4)	4*c*	0.4204(5)	1/4	0.4387(4)	0.017(2)	0.029(2)	0.033(2)	0	0.007(2)	0
O(5)	4*c*	0.6779(5)	1/4	0.3555(4)	0.016(2)	0.119(6)	0.021(2)	0	0.003(2)	0
O(6)	4*c*	0.0235(5)	1/4	0.3619(3)	0.018(2)	0.045(3)	0.018(2)	0	0.000(2)	0
O(7)	8*d*	0.3190(4)	0.3143(2)	0.6791(3)	0.051(2)	0.019(2)	0.028(2)	0.001(1)	–0.020(1)	–0.002(1)
N(1)	4*c*	0.5819(8)	1/4	0.4444(5)	0.037(3)	0.047(4)	0.030(3)	0	0.002(2)	0
C(1)	8*d*	–0.0848(5)	0.3629(2)	0.5367(3)	0.022(2)	0.018(2)	0.021(2)	0.003(2)	–0.000(1)	–0.002(2)
C(2)	8*d*	0.0333(5)	0.3884(2)	0.4517(3)	0.020(2)	0.020(2)	0.020(2)	0.002(2)	–0.003(1)	–0.002(2)
C(3)	8*d*	–0.0059(5)	0.4402(2)	0.3874(4)	0.022(2)	0.020(2)	0.024(2)	–0.001(2)	–0.002(2)	0.002(2)
C(4)	8*d*	–0.1658(5)	0.4695(2)	0.4047(4)	0.028(2)	0.017(2)	0.028(2)	0.003(2)	–0.008(2)	–0.002(2)
C(5)	8*d*	–0.2843(6)	0.4447(2)	0.4868(4)	0.023(2)	0.024(2)	0.032(2)	0.006(2)	–0.002(2)	–0.002(2)
C(6)	8*d*	–0.2460(6)	0.3927(2)	0.5509(4)	0.022(2)	0.025(2)	0.032(2)	0.005(2)	0.006(2)	0.002(2)
C(7)	8*d*	–0.1980(6)	0.5260(2)	0.3424(4)	0.035(2)	0.022(2)	0.030(2)	0.002(2)	–0.006(2)	0.002(2)
C(8)	8*d*	0.3075(6)	0.3778(2)	0.3551(5)	0.026(2)	0.037(3)	0.046(3)	0.003(2)	0.014(2)	0.018(2)

References

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2. Sheldrick, G. M.: SHELXL-97. Program for the Refinement of Crystal Structures. University of Göttingen, Germany 1997.