Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

Poly[[tetrakis(μ_2-pyrazine *N*,*N*′-dioxide-κ^2O:O')erbium(III)] tris(perchlorate)]

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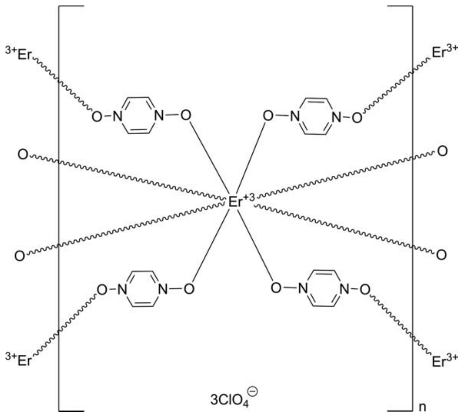
Received 3 August 2010; accepted 9 August 2010

Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; *R* factor = 0.032; *wR* factor = 0.092; data-to-parameter ratio = 18.2.

The title three-dimensional coordination network, $\{[Er(C_4H_4N_2O_2)_4](ClO_4)_3\}_n$, is isostructural to that of other lanthanides. The Er^{+3} cation lies on a fourfold roto-inversion axis. It is coordinated in a distorted square-antiprismatic fashion by eight O atoms from bridging pyrazine *N*,*N*′-dioxide ligands. There are two unique pyrazine *N*,*N*′-dioxide ligands. One ring is located around an inversion center, and there is a a twofold rotation axis at the center of the other ring. There are also two unique perchlorate anions. One is centered on a twofold rotation axis and the other on a fourfold roto-inversion axis. The perchlorate anions are located in channels that run perpendicular to (001) and (110) and interact with the coordination network through C—H⋯O hydrogen bonds.

Related literature

For the isostructural La, Ce, Pr, Sm, Eu, Gd, Tb and Y coordination networks, see: Sun *et al.* (2004). For the isostructural Nd, Dy and Ho coordination networks, see: Quinn-Elmore *et al.* (2010*a*,*b*); Buchner *et al.* (2010), respectively. Detailed background to this study is described in the first article of this series by Quinn-Elmore *et al.* (2010*a*).



Experimental

Crystal data

$[Er(C_4H_4N_2O_2)_4](ClO_4)_3$
M_r = 913.98
Tetragonal, $I4_1/acd$
a = 15.1777 (4) Å
c = 22.5094 (12) Å
V = 5185.3 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 3.66 mm^{-1}
T = 100 K
0.44 × 0.44 × 0.18 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.725, T_{max} = 1.000
27002 measured reflections
1997 independent reflections
1775 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.092
S = 0.98
1997 reflections
110 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 3.41 e Å^{-3}
$\Delta\rho_{min}$ = −1.62 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C2—H2⋯O2^{i}	0.95	2.50	3.266 (3)	138
C2—H2⋯O5	0.95	2.39	3.147 (5)	137
C3—H3⋯O1	0.95	2.60	3.321 (3)	133
C3—H3⋯O3	0.95	2.45	3.219 (3)	138
C4—H4⋯O3^{ii}	0.95	2.35	3.218 (3)	152

Symmetry codes: (i) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-y+\frac{3}{4}, x-\frac{1}{4}, -z+\frac{1}{4}$.

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *X-SEED*.

The authors are thankful to Allegheny College for providing funding in support of this research. The diffractometer was funded by the NSF (grant No. 0087210), the Ohio Board of Regents (grant No. CAP-491) and by Youngstown State University. The authors would like to acknowledge Youngstown State University and the STaRBURSTT CyberInstrumentation Consortium for assistance with the crystallography.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2301).

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supporting information

Acta Cryst. (2010). E**66**, m1110–m1111 [https://doi.org/10.1107/S1600536810031843]

Poly[[tetrakis(μ_2-pyrazine *N*,*N*′-dioxide-κ^2*O*:*O*′)erbium(III)] tris(perchlorate)]

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S1. Comment

The description of the structure of the title compound is part of a series of consecutive papers on three-dimensional coordination networks of the type $\{[Ln(C_4H_4N_2O_2)_4](ClO_4)_3\}_n$, with Ln = Nd (Quinn-Elmore *et al.* 2010*a*), Dy (Quinn-Elmore *et al.* 2010*b*) and Ho (Buchner *et al.* 2010), Er (this publication), respectively. All four compounds are also isostructural to the previously reported La, Ce, Pr, Sm, Eu, Gd, Tb and Y coordination networks (Sun *et al.* 2004). The background to this study is given in ther first article of this series by Quinn-Elmore *et al.* (2010*a*).

S2. Experimental

Pyrazine *N,N*′-dioxide (0.025 g, 0.223 mmol) was dissolved in deionized water (1.5 ml) and methanol (1.5 ml). An aqueous solution of $Er(ClO_4)_3$ (0.650 ml of a 0.0860 *M* solution, 0.0558 mmol) was diluted with methanol (0.350 ml) and CH_2Cl_2 (2.5 ml). The pyrazine *N,N*′-dioxide solution was layered over the $Er(ClO_4)_3$ solution, and the two solutions were allowed to slowly mix. Yellow block-like crystals formed upon the slow evaporation of the resultant solution.

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model with C—H = 0.95 Å and with U_{iso}(H) = 1.2 times U_{eq}(C).

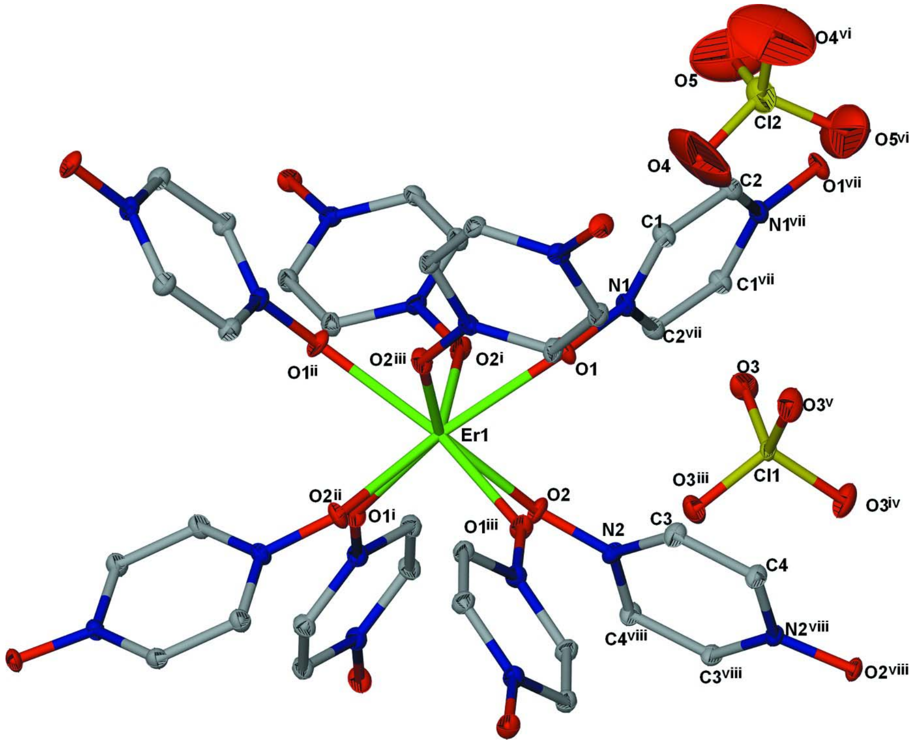

Figure 1

The coordination environment of the Er^{+3} cation in title compound with atom labels and 50% probability displacement ellipsoids. Hydrogen atoms have been omitted for clarity. Symmetry codes: (i) *y* + 1/4, *x* - 1/4, -*z* + 3/4; (ii) -*y* + 3/4, -*x* + 3/4, -*z* + 3/4; (iii) -*x* + 1, -*y* + 1/2, *z*; (iv) -*y* + 3/4, *x* - 1/4, -*z* + 1/4; (v) *y* + 1/4, -*x* + 3/4, -*z* + 1/4; (vi) *y* + 3/4, *x* - 3/4, -*z* + 1/4; (vii) -*x* + 3/2, -*y* + 1/2, -*z* + 1/2; (viii) *x*, -*y* + 1, -*z* + 1/2.

Poly[[tetrakis(μ_2-pyrazine *N*,*N′*-dioxide-κ^2*O*:*O′*)erbium(III)] tris(perchlorate)]

Crystal data

$[Er(C_4H_4N_2O_2)_4](ClO_4)_3$
$M_r = 913.98$
Tetragonal, *I*4$_1$/*acd*
Hall symbol: -I 4bd 2c
$a = 15.1777$ (4) Å
$c = 22.5094$ (12) Å
$V = 5185.3$ (3) Å^3
$Z = 8$
$F(000) = 3576$
$D_x = 2.342$ Mg m^{-3}
Mo *Kα* radiation, $\lambda = 0.71073$ Å
Cell parameters from 13828 reflections
$\theta = 2.6$–30.6°
$\mu = 3.66$ mm^{-1}
$T = 100$ K
Block, yellow
0.44 × 0.44 × 0.18 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.725$, $T_{max} = 1.000$
27002 measured reflections
1997 independent reflections
1775 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 30.6°$, $\theta_{min} = 2.6°$
$h = -21 \rightarrow 21$
$k = -21 \rightarrow 21$
$l = -32 \rightarrow 32$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.032$	H-atom parameters constrained
$wR(F^2) = 0.092$	$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 37.1849P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.98$	$(\Delta/\sigma)_{max} = 0.001$
1997 reflections	$\Delta\rho_{max} = 3.41$ e Å^{-3}
110 parameters	$\Delta\rho_{min} = -1.62$ e Å^{-3}
0 restraints	
Primary atom site location: structure-invariant direct methods	

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
Er1	0.5000	0.2500	0.3750	0.00421 (10)
Cl1	0.5000	0.2500	0.1250	0.0105 (3)
Cl2	0.72764 (5)	−0.02236 (5)	0.1250	0.0251 (2)
O1	0.59052 (11)	0.21840 (12)	0.29594 (8)	0.0150 (3)
O2	0.53202 (12)	0.39323 (11)	0.34440 (8)	0.0146 (3)
O3	0.57685 (14)	0.24307 (14)	0.16232 (9)	0.0230 (4)
O4	0.6459 (3)	−0.0156 (4)	0.1512 (3)	0.116 (2)
O5	0.7922 (3)	−0.0078 (3)	0.1690 (3)	0.0919 (17)
N1	0.66916 (14)	0.23390 (14)	0.27491 (9)	0.0130 (4)
N2	0.52617 (14)	0.44427 (13)	0.29795 (9)	0.0120 (4)
C1	0.70908 (16)	0.17164 (16)	0.24123 (10)	0.0142 (4)
H1	0.6810	0.1164	0.2351	0.017*
C2	0.78967 (15)	0.18759 (16)	0.21586 (11)	0.0140 (4)
H2	0.8168	0.1438	0.1918	0.017*
C3	0.52531 (17)	0.41139 (15)	0.24210 (10)	0.0138 (4)
H3	0.5249	0.3494	0.2361	0.017*
C4	0.52504 (17)	0.46718 (15)	0.19415 (10)	0.0142 (4)
H4	0.5240	0.4436	0.1551	0.017*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Er1	0.00435 (11)	0.00435 (11)	0.00392 (14)	−0.00025 (5)	0.000	0.000
Cl1	0.0123 (5)	0.0123 (5)	0.0067 (7)	0.000	0.000	0.000
Cl2	0.0235 (3)	0.0235 (3)	0.0283 (5)	−0.0089 (4)	0.0048 (2)	−0.0048 (2)

O1	0.0094 (7)	0.0220 (9)	0.0137 (7)	−0.0028 (6)	0.0040 (6)	−0.0033 (7)
O2	0.0220 (9)	0.0114 (7)	0.0105 (7)	−0.0015 (6)	−0.0032 (7)	0.0043 (6)
O3	0.0168 (9)	0.0376 (12)	0.0147 (9)	0.0044 (7)	−0.0043 (8)	−0.0022 (7)
O4	0.052 (3)	0.120 (4)	0.175 (5)	−0.003 (3)	0.075 (3)	0.000 (4)
O5	0.077 (3)	0.070 (3)	0.129 (5)	−0.002 (2)	−0.055 (3)	−0.029 (3)
N1	0.0110 (9)	0.0165 (9)	0.0113 (9)	−0.0011 (7)	0.0013 (7)	−0.0005 (8)
N2	0.0136 (9)	0.0107 (8)	0.0117 (9)	0.0001 (7)	−0.0016 (7)	0.0023 (7)
C1	0.0131 (10)	0.0149 (10)	0.0145 (10)	−0.0009 (8)	0.0016 (8)	−0.0019 (8)
C2	0.0122 (10)	0.0169 (11)	0.0128 (10)	−0.0003 (8)	0.0007 (8)	−0.0022 (8)
C3	0.0183 (11)	0.0105 (10)	0.0125 (10)	−0.0021 (8)	−0.0015 (9)	−0.0007 (8)
C4	0.0193 (11)	0.0115 (10)	0.0118 (10)	−0.0002 (8)	−0.0002 (9)	−0.0002 (8)

Geometric parameters (Å, º)

Er1—O1$^{\text{i}}$	2.2989 (17)	Cl2—O5$^{\text{vi}}$	1.410 (4)
Er1—O1$^{\text{ii}}$	2.2989 (17)	O1—N1	1.305 (3)
Er1—O1	2.2989 (17)	O2—N2	1.304 (2)
Er1—O1$^{\text{iii}}$	2.2989 (17)	N1—C1	1.354 (3)
Er1—O2$^{\text{i}}$	2.3316 (17)	N1—C2$^{\text{vii}}$	1.361 (3)
Er1—O2$^{\text{ii}}$	2.3316 (17)	N2—C3	1.353 (3)
Er1—O2	2.3316 (17)	N2—C4$^{\text{viii}}$	1.356 (3)
Er1—O2$^{\text{iii}}$	2.3316 (17)	C1—C2	1.371 (3)
Cl1—O3	1.441 (2)	C1—H1	0.9500
Cl1—O3$^{\text{iv}}$	1.441 (2)	C2—N1$^{\text{vii}}$	1.361 (3)
Cl1—O3$^{\text{iii}}$	1.441 (2)	C2—H2	0.9500
Cl1—O3$^{\text{v}}$	1.441 (2)	C3—C4	1.372 (3)
Cl2—O4$^{\text{vi}}$	1.377 (4)	C3—H3	0.9500
Cl2—O4	1.377 (4)	C4—N2$^{\text{viii}}$	1.356 (3)
Cl2—O5	1.410 (4)	C4—H4	0.9500

O1$^{\text{i}}$—Er1—O1$^{\text{ii}}$	78.54 (9)	O3$^{\text{iv}}$—Cl1—O3$^{\text{iii}}$	109.86 (9)
O1$^{\text{i}}$—Er1—O1	148.06 (9)	O3—Cl1—O3$^{\text{v}}$	109.86 (9)
O1$^{\text{ii}}$—Er1—O1	110.48 (9)	O3$^{\text{iv}}$—Cl1—O3$^{\text{v}}$	108.69 (17)
O1$^{\text{i}}$—Er1—O1$^{\text{iii}}$	110.48 (9)	O3$^{\text{iii}}$—Cl1—O3$^{\text{v}}$	109.86 (9)
O1$^{\text{ii}}$—Er1—O1$^{\text{iii}}$	148.06 (9)	O4$^{\text{vi}}$—Cl2—O4	108.5 (5)
O1—Er1—O1$^{\text{iii}}$	78.55 (9)	O4$^{\text{vi}}$—Cl2—O5	113.0 (3)
O1$^{\text{i}}$—Er1—O2$^{\text{i}}$	80.86 (7)	O4—Cl2—O5	108.3 (4)
O1$^{\text{ii}}$—Er1—O2$^{\text{i}}$	72.63 (6)	O4$^{\text{vi}}$—Cl2—O5$^{\text{vi}}$	108.3 (4)
O1—Er1—O2$^{\text{i}}$	73.44 (6)	O4—Cl2—O5$^{\text{vi}}$	113.0 (3)
O1$^{\text{iii}}$—Er1—O2$^{\text{i}}$	137.94 (6)	O5—Cl2—O5$^{\text{vi}}$	105.9 (5)
O1$^{\text{i}}$—Er1—O2$^{\text{ii}}$	72.63 (6)	N1—O1—Er1	142.15 (15)
O1$^{\text{ii}}$—Er1—O2$^{\text{ii}}$	80.86 (7)	N2—O2—Er1	140.93 (15)
O1—Er1—O2$^{\text{ii}}$	137.94 (6)	O1—N1—C1	119.1 (2)
O1$^{\text{iii}}$—Er1—O2$^{\text{ii}}$	73.44 (6)	O1—N1—C2$^{\text{vii}}$	121.5 (2)
O2$^{\text{i}}$—Er1—O2$^{\text{ii}}$	145.63 (8)	C1—N1—C2$^{\text{vii}}$	119.4 (2)
O1$^{\text{i}}$—Er1—O2	73.44 (6)	O2—N2—C3	121.8 (2)
O1$^{\text{ii}}$—Er1—O2	137.94 (6)	O2—N2—C4$^{\text{viii}}$	119.0 (2)
O1—Er1—O2	80.86 (7)	C3—N2—C4$^{\text{viii}}$	119.2 (2)

O1^{iii}—Er1—O2	72.63 (6)	N1—C1—C2	120.6 (2)
O2^{i}—Er1—O2	72.46 (9)	N1—C1—H1	119.7
O2^{ii}—Er1—O2	118.42 (9)	C2—C1—H1	119.7
O1^{i}—Er1—O2^{iii}	137.94 (6)	N1^{vii}—C2—C1	120.0 (2)
O1^{ii}—Er1—O2^{iii}	73.44 (6)	N1^{vii}—C2—H2	120.0
O1—Er1—O2^{iii}	72.63 (6)	C1—C2—H2	120.0
O1^{iii}—Er1—O2^{iii}	80.87 (7)	N2—C3—C4	120.2 (2)
O2^{i}—Er1—O2^{iii}	118.42 (9)	N2—C3—H3	119.9
O2^{ii}—Er1—O2^{iii}	72.46 (9)	C4—C3—H3	119.9
O2—Er1—O2^{iii}	145.64 (8)	N2^{viii}—C4—C3	120.6 (2)
O3—Cl1—O3^{iv}	109.86 (9)	N2^{viii}—C4—H4	119.7
O3—Cl1—O3^{iii}	108.69 (17)	C3—C4—H4	119.7

Symmetry codes: (i) *y*+1/4, *x*−1/4, −*z*+3/4; (ii) −*y*+3/4, −*x*+3/4, −*z*+3/4; (iii) −*x*+1, −*y*+1/2, *z*; (iv) −*y*+3/4, *x*−1/4, −*z*+1/4; (v) *y*+1/4, −*x*+3/4, −*z*+1/4; (vi) *y*+3/4, *x*−3/4, −*z*+1/4; (vii) −*x*+3/2, −*y*+1/2, −*z*+1/2; (viii) *x*, −*y*+1, −*z*+1/2.

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Symmetry codes: (iv) −*y*+3/4, *x*−1/4, −*z*+1/4; (vii) −*x*+3/2, −*y*+1/2, −*z*+1/2.