

## Poly[[tetrakis( $\mu_2$ -pyrazine $N,N'$ -dioxide- $\kappa^2O:O'$ )erbium(III)] tris(perchlorate)]

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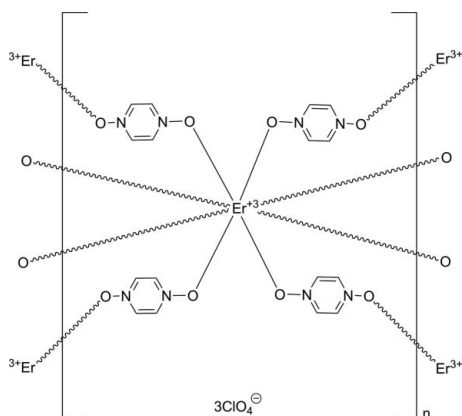
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Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.032;  $wR$  factor = 0.092; data-to-parameter ratio = 18.2.

The title three-dimensional coordination network,  $\{[\text{Er}(\text{C}_4\text{H}_4\text{N}_2\text{O}_2)_4](\text{ClO}_4)_3\}_n$ , is isostructural to that of other lanthanides. The  $\text{Er}^{+3}$  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 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  $\text{C}-\text{H}\cdots\text{O}$  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

$[\text{Er}(\text{C}_4\text{H}_4\text{N}_2\text{O}_2)_4](\text{ClO}_4)_3$   
 $M_r = 913.98$   
 Tetragonal,  $I4_1/acd$   
 $a = 15.1777$  (4) Å  
 $c = 22.5094$  (12) Å  
 $V = 5185.3$  (3) Å<sup>3</sup>

$Z = 8$   
 Mo  $K\alpha$  radiation  
 $\mu = 3.66$  mm<sup>-1</sup>  
 $T = 100$  K  
 $0.44 \times 0.44 \times 0.18$  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  
 1977 independent reflections  
 1775 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.024$

#### 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  
 $\Delta\rho_{\text{max}} = 3.41$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -1.62$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}2-\text{H}2\cdots\text{O}2^{\text{i}}$	0.95	2.50	3.266 (3)	138
$\text{C}2-\text{H}2\cdots\text{O}5$	0.95	2.39	3.147 (5)	137
$\text{C}3-\text{H}3\cdots\text{O}1$	0.95	2.60	3.321 (3)	133
$\text{C}3-\text{H}3\cdots\text{O}3$	0.95	2.45	3.219 (3)	138
$\text{C}4-\text{H}4\cdots\text{O}3^{\text{ii}}$	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.

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

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**supplementary materials**

*Acta Cryst.* (2010). E66, m1110-m1111 [ doi:10.1107/S1600536810031843 ]

**Poly[[tetrakis( $\mu_2$ -pyrazine *N,N'*-dioxide- $\kappa^2O:O'$ )erbium(III)] tris(perchlorate)]**

**J. D. Buchner, B. G. Quinn-Elmore, K. B. Beach and J. M. Knaust**

**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  $\{[\text{Ln}(\text{C}_4\text{H}_4\text{N}_2\text{O}_2)_4](\text{ClO}_4)_3\}_n$ , 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 their first article of this series by Quinn-Elmore *et al.* (2010*a*).

**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  $\text{Er}(\text{ClO}_4)_3$  (0.650 ml of a 0.0860 M solution, 0.0558 mmol) was diluted with methanol (0.350 ml) and  $\text{CH}_2\text{Cl}_2$  (2.5 ml). The pyrazine *N,N'*-dioxide solution was layered over the  $\text{Er}(\text{ClO}_4)_3$  solution, and the two solutions were allowed to slowly mix. Yellow block-like crystals formed upon the slow evaporation of the resultant solution.

**Refinement**

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

**Figures**

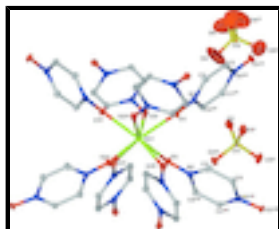


Fig. 1. The coordination environment of the  $\text{Er}^{+3}$  cation in title compound with atom labels and 50% probability displacement ellipsoids. Hydrogen atoms have been omitted for clarity. Symmetry codes: (i)  $y + 1/4, x - 1/4, -z + 3/4$ ; (ii)  $-y + 3/4, -x + 3/4, -z + 3/4$ ; (iii)  $-x + 1, -y + 1/2, z$ ; (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( $\mu_2$ -pyrazine *N,N'*-dioxide- $\kappa^2O:O'$ )erbium(III)] tris(perchlorate)]**

*Crystal data*

$[\text{Er}(\text{C}_4\text{H}_4\text{N}_2\text{O}_2)_4](\text{ClO}_4)_3$

$M_r = 913.98$

Tetragonal,  $I4_1/acd$

Hall symbol: -I 4bd 2c

$D_x = 2.342 \text{ Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 13828 reflections

$\theta = 2.6\text{--}30.6^\circ$

# supplementary materials

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$a = 15.1777 (4) \text{ \AA}$	$\mu = 3.66 \text{ mm}^{-1}$
$c = 22.5094 (12) \text{ \AA}$	$T = 100 \text{ K}$
$V = 5185.3 (3) \text{ \AA}^3$	Block, yellow
$Z = 8$	$0.44 \times 0.44 \times 0.18 \text{ mm}$
$F(000) = 3576$	

## Data collection

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

## Refinement

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

## 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 ( $\text{\AA}^2$ )

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
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 ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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 ( $\text{\AA}$ ,  $^\circ$ )

Er1—O1 <sup>i</sup>	2.2989 (17)	Cl2—O5 <sup>vi</sup>	1.410 (4)
Er1—O1 <sup>ii</sup>	2.2989 (17)	O1—N1	1.305 (3)
Er1—O1	2.2989 (17)	O2—N2	1.304 (2)
Er1—O1 <sup>iii</sup>	2.2989 (17)	N1—C1	1.354 (3)
Er1—O2 <sup>i</sup>	2.3316 (17)	N1—C2 <sup>vii</sup>	1.361 (3)
Er1—O2 <sup>ii</sup>	2.3316 (17)	N2—C3	1.353 (3)
Er1—O2	2.3316 (17)	N2—C4 <sup>viii</sup>	1.356 (3)
Er1—O2 <sup>iii</sup>	2.3316 (17)	C1—C2	1.371 (3)
Cl1—O3	1.441 (2)	C1—H1	0.9500

## supplementary materials

Cl1—O3 <sup>iv</sup>	1.441 (2)	C2—N1 <sup>vii</sup>	1.361 (3)
Cl1—O3 <sup>iii</sup>	1.441 (2)	C2—H2	0.9500
Cl1—O3 <sup>v</sup>	1.441 (2)	C3—C4	1.372 (3)
Cl2—O4 <sup>vi</sup>	1.377 (4)	C3—H3	0.9500
Cl2—O4	1.377 (4)	C4—N2 <sup>viii</sup>	1.356 (3)
Cl2—O5	1.410 (4)	C4—H4	0.9500
O1 <sup>i</sup> —Er1—O1 <sup>ii</sup>	78.54 (9)	O3 <sup>iv</sup> —Cl1—O3 <sup>iii</sup>	109.86 (9)
O1 <sup>i</sup> —Er1—O1	148.06 (9)	O3—Cl1—O3 <sup>v</sup>	109.86 (9)
O1 <sup>ii</sup> —Er1—O1	110.48 (9)	O3 <sup>iv</sup> —Cl1—O3 <sup>v</sup>	108.69 (17)
O1 <sup>i</sup> —Er1—O1 <sup>iii</sup>	110.48 (9)	O3 <sup>iii</sup> —Cl1—O3 <sup>v</sup>	109.86 (9)
O1 <sup>ii</sup> —Er1—O1 <sup>iii</sup>	148.06 (9)	O4 <sup>vi</sup> —Cl2—O4	108.5 (5)
O1—Er1—O1 <sup>iii</sup>	78.55 (9)	O4 <sup>vi</sup> —Cl2—O5	113.0 (3)
O1 <sup>i</sup> —Er1—O2 <sup>i</sup>	80.86 (7)	O4—Cl2—O5	108.3 (4)
O1 <sup>ii</sup> —Er1—O2 <sup>i</sup>	72.63 (6)	O4 <sup>vi</sup> —Cl2—O5 <sup>vi</sup>	108.3 (4)
O1—Er1—O2 <sup>i</sup>	73.44 (6)	O4—Cl2—O5 <sup>vi</sup>	113.0 (3)
O1 <sup>iii</sup> —Er1—O2 <sup>i</sup>	137.94 (6)	O5—Cl2—O5 <sup>vi</sup>	105.9 (5)
O1 <sup>i</sup> —Er1—O2 <sup>ii</sup>	72.63 (6)	N1—O1—Er1	142.15 (15)
O1 <sup>ii</sup> —Er1—O2 <sup>ii</sup>	80.86 (7)	N2—O2—Er1	140.93 (15)
O1—Er1—O2 <sup>ii</sup>	137.94 (6)	O1—N1—C1	119.1 (2)
O1 <sup>iii</sup> —Er1—O2 <sup>ii</sup>	73.44 (6)	O1—N1—C2 <sup>vii</sup>	121.5 (2)
O2 <sup>i</sup> —Er1—O2 <sup>ii</sup>	145.63 (8)	C1—N1—C2 <sup>vii</sup>	119.4 (2)
O1 <sup>i</sup> —Er1—O2	73.44 (6)	O2—N2—C3	121.8 (2)
O1 <sup>ii</sup> —Er1—O2	137.94 (6)	O2—N2—C4 <sup>viii</sup>	119.0 (2)
O1—Er1—O2	80.86 (7)	C3—N2—C4 <sup>viii</sup>	119.2 (2)
O1 <sup>iii</sup> —Er1—O2	72.63 (6)	N1—C1—C2	120.6 (2)
O2 <sup>i</sup> —Er1—O2	72.46 (9)	N1—C1—H1	119.7
O2 <sup>ii</sup> —Er1—O2	118.42 (9)	C2—C1—H1	119.7
O1 <sup>i</sup> —Er1—O2 <sup>iii</sup>	137.94 (6)	N1 <sup>vii</sup> —C2—C1	120.0 (2)
O1 <sup>ii</sup> —Er1—O2 <sup>iii</sup>	73.44 (6)	N1 <sup>vii</sup> —C2—H2	120.0
O1—Er1—O2 <sup>iii</sup>	72.63 (6)	C1—C2—H2	120.0
O1 <sup>iii</sup> —Er1—O2 <sup>iii</sup>	80.87 (7)	N2—C3—C4	120.2 (2)
O2 <sup>i</sup> —Er1—O2 <sup>iii</sup>	118.42 (9)	N2—C3—H3	119.9
O2 <sup>ii</sup> —Er1—O2 <sup>iii</sup>	72.46 (9)	C4—C3—H3	119.9
O2—Er1—O2 <sup>iii</sup>	145.64 (8)	N2 <sup>viii</sup> —C4—C3	120.6 (2)
O3—Cl1—O3 <sup>iv</sup>	109.86 (9)	N2 <sup>viii</sup> —C4—H4	119.7
O3—Cl1—O3 <sup>iii</sup>	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)  $x, -y+1, -z+1/2$ .

### Hydrogen-bond geometry ( $\text{\AA}, ^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
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C2—H2···O2 <sup>vii</sup>	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 <sup>iv</sup>	0.95	2.35	3.218 (3)	152.

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



Fig. 1

