

Zhen-Zhong Lu,^a Shan Gao^a and
Seik Weng Ng^{b*}^aCollege of Chemistry and Materials Science,
Heilongjiang University, Harbin 150080,
People's Republic of China, and ^bDepartment of
Chemistry, University of Malaya, Kuala Lumpur
50603, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Key indicators

Single-crystal X-ray study
 $T = 295$ K
Mean $\sigma(\text{O}-\text{C}) = 0.006$ Å
Disorder in main residue
 R factor = 0.039
 wR factor = 0.089
Data-to-parameter ratio = 12.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**catena-Poly[[diaqua(2-chloronicotinato- $\kappa^2\text{O},\text{O}'$)-
erbium(III)]-di- μ -2-chloronicotinato- $\kappa^4\text{O}:\text{O}'$]**

The asymmetric unit of the title compound, $[\text{Er}(\text{C}_6\text{H}_3\text{ClNO}_2)_3(\text{H}_2\text{O})_2]_n$, has two independent disordered 2-chloronicotinate groups, one of which lies on a twofold rotation axis. The group lying on this symmetry element chelates to the water-coordinated Er atom, which also lies on the rotation axis. Two more 2-chloronicotinate groups bridge two diaqua(2-chloronicotinato)erbium species into a linear chain. The Er atom exists in a square-antiprismatic environment made up of O atoms.

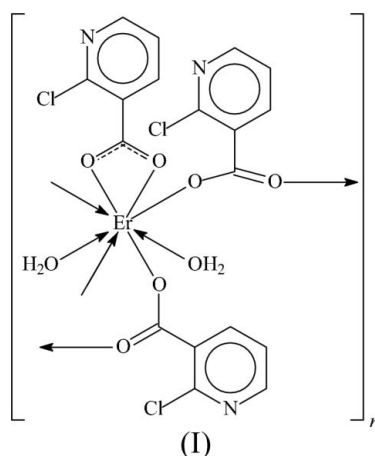
Received 27 September 2005

Accepted 14 October 2005

Online 19 October 2005

Comment

Lanthanide derivatives of the three isomeric pyridylcarboxylic acids represent a class of metal pyridylcarboxylates for which there are relatively few structurally authenticated examples (Mao *et al.*, 1998). With erbium(III), a trinicotinate has been isolated as a dihydrate (Baker, Cook, Hutchison, Leask *et al.*, 1991; Baker, Cook, Hutchison, Martineau *et al.*, 1991), but its crystal structure has not been reported to date. The erbium(III) derivative of 2-chloronicotinic acid exists as the title diaqua compound, (I) (Fig. 1).



The formula unit of (I) has one 2-chloronicotinate anion that chelates to the metal atom; the other two behave as bridging ligands to give rise to a linear chain motif (Fig. 2). The metal atom, located on a twofold axis, exists in a square-antiprismatic coordination polyhedron by O atoms (Fig. 3).

Hydrogen bonds link adjacent chains into a three-dimensional network structure (Table 2).

Experimental

Erbium trichloride hexahydrate (0.76 g, 2 mmol) and 2-chloronicotinic acid (0.95 g, 6 mmol) were dissolved in a small volume of

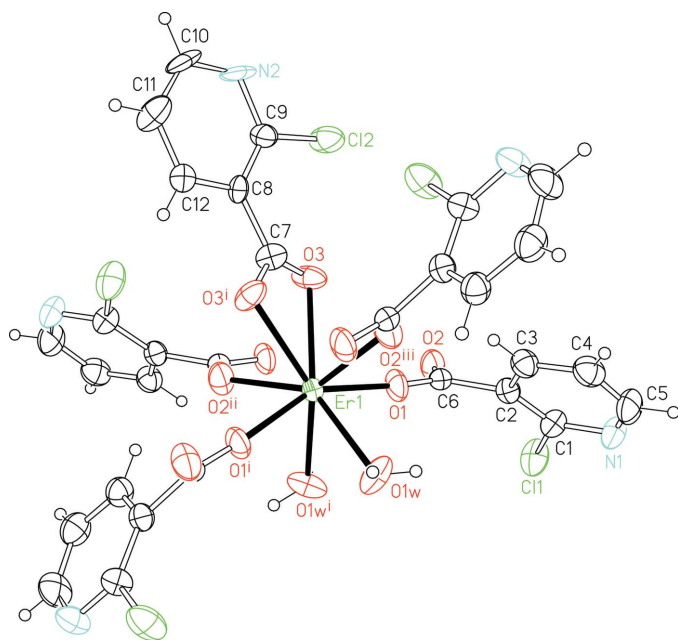


Figure 1
A plot of a portion of the chain structure of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Symmetry codes are as given in Table 1.

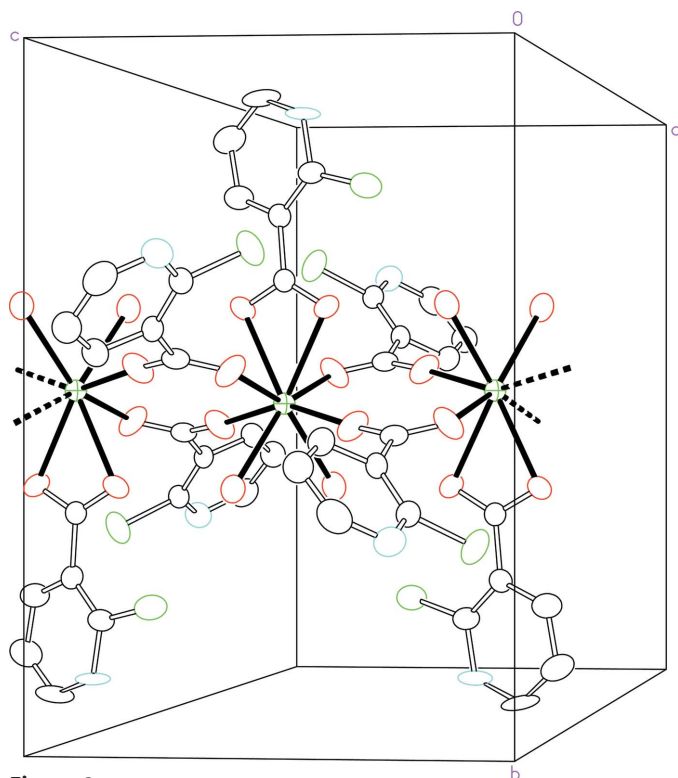


Figure 2
A diagram of the chain structure. H atoms have been omitted.

water. Aqueous sodium hydroxide solution (0.1 M) was added dropwise until the pH of the solution was about 5. The solution was filtered and set aside for a month for the growth of pink prismatic crystals of (I). CHN analysis, calculated for $C_{18}H_{13}Cl_3N_3O_8Er$: C 32.13, H 1.95, N 6.24%; found: C 32.16, H 1.92, N 6.21%.

Crystal data

$[Er(C_6H_3ClNO_2)_3(H_2O)_2]$
 $M_r = 672.92$
 Monoclinic, $C2/c$
 $a = 16.324(3) \text{ \AA}$
 $b = 14.130(3) \text{ \AA}$
 $c = 9.614(2) \text{ \AA}$
 $\beta = 104.05(3)^\circ$
 $V = 2151.2(8) \text{ \AA}^3$
 $Z = 4$

$D_x = 2.078 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 Cell parameters from 9329 reflections
 $\theta = 3.1\text{--}27.5^\circ$
 $\mu = 4.33 \text{ mm}^{-1}$
 $T = 295(2) \text{ K}$
 Prism, pink
 $0.10 \times 0.10 \times 0.10 \text{ mm}$

Data collection

Rigaku R-Axis RAPID IP diffractometer
 ω scans
 Absorption correction: multi-scan (ABSCOR; Higashi, 1995)
 $T_{\min} = 0.170, T_{\max} = 0.672$
 10139 measured reflections

2464 independent reflections
 2317 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.066$
 $\theta_{\max} = 27.5^\circ$
 $h = -21 \rightarrow 21$
 $k = -18 \rightarrow 18$
 $l = -11 \rightarrow 12$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.089$
 $S = 1.14$
 2464 reflections
 205 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.035P)^2 + 5.2256P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.60 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -1.81 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters ($\text{\AA}, ^\circ$).

Er1—O1	2.237 (3)	Er1—O3	2.450 (4)
Er1—O2 ⁱ	2.330 (3)	Er1—O1W	2.381 (4)
O1—Er1—O1 ⁱⁱ	154.3 (2)	O2 ⁱ —Er1—O3	72.4 (1)
O1—Er1—O2 ⁱⁱ	106.5 (1)	O2 ⁱ —Er1—O3 ⁱⁱⁱ	75.3 (2)
O1—Er1—O2 ⁱⁱⁱ	81.6 (1)	O2 ⁱ —Er1—O1W	140.4 (2)
O1—Er1—O3	77.5 (1)	O2 ⁱ —Er1—O1W ⁱⁱ	74.4 (2)
O1—Er1—O3 ⁱⁱ	128.0 (1)	O3—Er1—O3 ⁱⁱⁱ	53.0 (2)
O1—Er1—O1W	84.9 (2)	O3—Er1—O1W	146.8 (1)
O1—Er1—O1W ⁱⁱ	74.3 (1)	O3—Er1—O1W ⁱⁱ	127.3 (1)
O2 ⁱ —Er1—O2 ⁱⁱⁱ	143.8 (2)	O1W—Er1—O1W ⁱⁱ	72.6 (2)

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

Table 2

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—H1W1 \cdots N1 ^{iv}	0.82	2.24	2.87 (3)	134
O1W—H1W1 \cdots N1 ^{iv}	0.82	2.34	2.95 (3)	132
O1W—H1W2 \cdots O3 ⁱⁱⁱ	0.82	2.02	2.776 (5)	153

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

Both 2-chloronicotinate groups, one lying on a general position and the other on the twofold rotation axis, are disordered. For that on a general position, the disorder affects the pyridyl portion but not the carboxylate $-\text{CO}_2$ portion. The bond distances between the two portions were restrained to within 0.01 \AA of each other; pairs of distances (e.g. C1—C1 and C1'—C1', C1—C2 and C1'—C2', etc.) were similarly restrained. The pyridyl rings were restrained to be nearly planar, and the displacement parameters of the primed atoms were set to those of the unprimed atoms. The displacement parameters of the C atoms were restrained to be nearly isotropic. The occupancies refined to 53 (1):47 (1) for the unprimed and primed groups, respectively. For the 2-chloronicotinate group lying on the

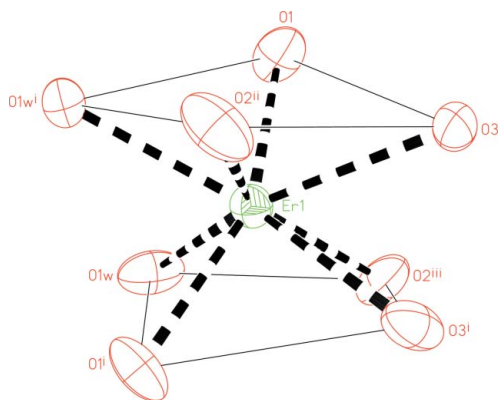


Figure 3
A diagram illustrating the eight-coordinate geometry of Er. Symmetry codes are as given in Table 1.

twofold rotation axis, the chloropyridyl portion was allowed to refine over this symmetry element with no atoms constrained to lie on the rotation axis. All atoms in this portion had half-site occupancy. H atoms were positioned geometrically, with $C-H = 0.93 \text{ \AA}$ and $U_{iso}(H) = 1.2U_{eq}(C)$, and were included in the refinement in the riding-model approximation. The two water H atoms were also positioned geometrically, with $O-H = 0.82 \text{ \AA}$ and $U_{iso}(H) = 1.2U_{eq}(O)$ and refined as riding. The final difference Fourier map had a large peak/hole at about 1 \AA from Er1.

Data collection: *RAPID AUTO* (Rigaku, 1998); cell refinement: *RAPID AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP II* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

The authors thank the National Natural Science Foundation of China (grant No. 20101003), the Scientific Fund for Remarkable Teachers of Heilongjiang Province (grant No. 1054 G036) and the University of Malaya for supporting this study.

References

- Baker, J. M., Cook, M. I., Hutchison, C. A., Leask, M. J. M., Robinson, M. G., Tronconi, A. L. & Wells, M. R. (1991). *Proc. R. Soc. London, Ser. A*, **434**, 695–706.
- Baker, J. M., Cook, M. I., Hutchison, C. A., Martineau, P. M., Tronconi, A. L. & Weber, R. T. (1991). *Proc. R. Soc. London, Ser. A*, **434**, 707–717.
- Higashi, T. (1995). *ABSCOR*. Program for Absorption Correction, Tokyo, Japan.
- Johnson, C. K. (1976). *ORTEP II*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Mao, J.-G., Zhang, H.-J., Ni, J.-Z., Wang, S.-B. & Mak, T. C. W. (1998). *Polyhedron*, **17**, 3999–4009.
- Rigaku (1998). *RAPID AUTO*. Rigaku Corporation, Tokyo, Japan.
- Rigaku/MS (2002). *CrystalStructure*. Version 3.00. Rigaku/MS, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.