Crystal structures of two erbium(III) complexes with 4-aminobenzoic acid and 4-chloro-3-nitrobenzoic acid

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Graham Smitha* and Daniel E. Lyncht

The crystal structures of two erbium(III) complexes with 4-aminobenzoic acid (4-ABAH), namely bis(μ₂-4-aminobenzoato-κ²O,O’)bis[4-aminobenzoato-κ²O,O’]diaquaerbium(III) dihydrate, [Er₂(C₇H₆NO₂)₆(H₂O)₄]·2H₂O, (I), and 4-chloro-3-nitrobenzoic acid (CLNBAH), namely poly[hexakis(μ₂-4-chloro-3-nitrobenzoato-κ²O,O’)bis(dimethyl sulfoxide-κO)dietherbium(III)], [Er₂(C₇H₃ClNO₄)₆(C₂H₆OS)₂]n, (II), have been determined. In the structure of solvatomorphic compound (I), the symmetry-related irregular ErO₈ coordination polyhedra in the discrete centrosymmetric dinuclear complex comprise two monodentate water molecules and six carboxylate O-atom donors, four from two bidentate carboxylate O₂O₂-chelate groups and two from the bis-monodentate O₂O₂-bridging group of the third 4-ABA anion. The Er—O bond-length range is 2.232 (3)–2.478 (3) Å and the Er···Er separation in the dinuclear complex unit is 4.7527 (4) Å. One of the coordinating water molecules is involved in an intra-unit O—H···O hydrogen-bonding association with an inversion-related carboxylate O-atom acceptor. In contrast, the anhydrous compound (II) is polymeric, based on centrosymmetric dinuclear repeat units comprising ErO₇ coordination polyhedra which involve four O-atom donors from two bidentate O₂O₂-bridging carboxylate groups, one O-atom donor from the monodentate dimethyl sulfoxide ligand and two O-atom donors from the third bridging CLNBA anion. The latter provides the inter-unit link in the one-dimensional coordination polymer extending along [100]. The Er···O bond-length range in (II) is 2.239 (6)–2.348 (6) Å and the Er···Er separation within the dinuclear unit is 4.4620 (6) Å. In the crystal of (I), extensive inter-dimer O—H···O and N—H···O hydrogen-bonding interactions involving both the coordinating water molecules and the solvent water molecules, as well as the amine groups of the 4-ABA anions, give an overall three-dimensional network structure. Within this structure are also weak π···π ring interactions between two of the coordinating ligands [ring-centroid separations = 3.676 (3) and 3.711 (2) Å]. With (II), only weak intra-polymer C—H···O, C—H···Cl and C—H···S interactions are present.

1. Chemical context

The coordination chemistry of the rare earth (RE) metals has been investigated extensively and the structures of a large number of complexes with various ligand types are known (Sastri et al., 2003). Of interest is the lanthanide contraction across the series and 4-aminobenzoic acid (4-ABAH) has provided a valuable ligand for this purpose in a comprehensive study of this effect with the RE³⁺ (La–Y) series of complexes (Sun et al., 2004). Within this series there are two sub-sets of isotypic complexes, one monoclinic (P2₁/n) (La–Tb as well as Dy and Er), in which the structures are two-dimensional, the second triclinic (P-T) forming dinuclear structures (Yb, Lu, Y, as well as Tb). The solvatomorphism of
the Tb member \{monoclinic, \[\text{Tb}_2(4\text{-ABA})_6(\text{H}_2\text{O})_2]\}; triclinic \[[\text{Tb}_2(4\text{-ABA})_6(\text{H}_2\text{O})_2] \cdot \text{H}_2\text{O}\}\] is of interest and its occurrence was indicated as being dependent on pH control in the preparation.

It was considered that some of the other later members of the RE series (predominantly triclinic) might also show the same effect so this was tested with Er in a reaction of erbium(III) acetate with 4-ABA in aqueous ethanol under mild reaction conditions, with no additional pH control. The title triclinic complex \[[\text{Er}_2(\text{C}_7\text{H}_6\text{NO}_2)_6(\text{H}_2\text{O})_4] \cdot \text{H}_2\text{O}\], (I), was obtained. For (I), the preliminary unit-cell data (Table 1) suggested a possible solvatomorphic variant of the previously reported polymeric monoclinic Er\textsuperscript{III} complex \[[\text{Er}_2(4\text{-ABA})_6(\text{H}_2\text{O})_2] \cdot \text{H}_2\text{O}\] (Sun \textit{et al.}, 2004), and this was confirmed in the X-ray structural analysis. The comparative cell data for the triclinic Tb\textsuperscript{III} complex with 4-ABA are

| Parameter          | Value      |
|--------------------|------------|
| \(a\)              | 9.0964     |
| \(b\)              | 11.0117    |
| \(c\)              | 12.7430    |
| \(\alpha\)         | 89.372     |
| \(\beta\)          | 72.0360    |
| \(\gamma\)         | 75.0730    |
| \(V\)              | 1169.97    |

Table 1: Selected bond lengths (Å) for (I).

| Bond Pair   | Distance (Å)   |
|-------------|----------------|
| Er\textsuperscript{I}—O\textsubscript{1}\textsuperscript{W} | 2.373 (2) |
| Er\textsuperscript{I}—O\textsubscript{1}\textsuperscript{2}\textsuperscript{A} | 2.333 (3) |
| Er\textsuperscript{I}—O\textsubscript{2}\textsuperscript{W} | 2.295 (3) |
| Er\textsuperscript{I}—O\textsubscript{1}\textsuperscript{2}\textsuperscript{B} | 2.385 (3) |
| Er\textsuperscript{I}—O\textsubscript{1}\textsuperscript{1}\textsuperscript{A} | 2.477 (3) |
| Er\textsuperscript{I}—O\textsubscript{1}\textsuperscript{1}\textsuperscript{C} | 2.232 (3) |
| Er\textsuperscript{I}—O\textsubscript{1}\textsuperscript{1}\textsuperscript{B} | 2.478 (3) |

Symmetry code: (i) = \(-x, y, -z, -z + 1\).

subsequent recrystallization using DMSO. The structures of both complexes are reported herein.

2. Structural commentary

In the title centrosymmetric dinuclear structure of compound (I) (Fig. 1), the two identical irregular Er\textsuperscript{III} complex units \[[\text{Er}—\text{O bond length range, 2.232 (3)—2.478 (3) Å}]\] (Table 1), comprise two monodentate water molecules (O\textsubscript{1}W, O\textsubscript{2}W), four O-atom donors from two slightly asymmetric bidentate \(O,O'\) chelate carboxylate groups (the \(A\) and \(B\) 4-ABA ligands) and two bridging O-atom donors from two symmetry-related ligands (C). The Er\textsuperscript{I}—Er\textsuperscript{I} separation in the dinuclear unit is 4.7527 (4) Å. Unlike the polymeric solvatomorphic Er\textsuperscript{III} complex \[[\text{Er}_2(4\text{-ABA})_6(\text{H}_2\text{O})_2] \cdot \text{H}_2\text{O}\] (Sun \textit{et al.}, 2004), in which the extending Er—N bond is somewhat elongated at 2.660 (3) Å, with (I), there is no reasonable Er—N bonding contact. The monodentate water molecule O2W in (I) replaces the bridging amino N-donor site which is present in the 8-coordination sphere about Er in the solvatopolymer. Within the dinuclear complex unit of (I), an intra-dimer O—H\cdot\cdot\cdotO carboxylate hydrogen bond is present between one of the coordinating water molecules (O1W) and an inversion-
related carboxylate O-atom (O11A') (Table 2). This structure is similar to the triclinic isotypic Tb$^{3+}$ complex with 4-ABA (Sun et al., 2004).

In (I), the 4-ABA ligand species show some variation in the conformation of the carboxylate groups. In one of the bidentate $O,O'$-chelate ligands (A) and the bridging ligand (C), the groups are essentially coplanar with the benzene ring [torsion angles C2A/C—C1A/C—C11A/C—O11A/C = 171.2 (4) and 174.8 (4)$^\circ$, respectively], while in the second bidentate chelate ligand (B) the group is rotated out of the plane [corresponding torsion angle = 155.9 (4)$^\circ$]. Such a 'planar' conformation is also found in the structure of the parent acid (Gracin & Fischer, 2005) and in molecular adducts with aromatic carboxylic acids (Chadwick et al., 2009).

In the crystal structure of complex (II), a centro symmetric dinuclear repeat unit is present with the two inversion-related Er$^{III}$ atoms (Fig. 2) being seven-coordinated through four bridging carboxylate $O,O'$ groups (the A and B ligands), a monodentate DMSO O-atom and O-donors (O12C$^i$) and O11C$^i$ from the C ligand which extends the dinuclear unit into a one-dimensional coordination polymer lying along [100] (Fig. 3). The Er—O bond length range is 2.239 (6)–2.348 (6) Å (Table 3) and the Er–·Er separation in the dimeric unit is 4.4620 (6) Å. Also present within the repeat unit are a C2B—H—·O11 hydrogen bond [3.298 (13) Å] and a C2A—H—·S1 interaction [3.743 (10) Å] (Table 4).

The torsion angles defining the conformation of the carboxylate groups of the CLNBA ligands in (II) are C2A/B/C—C1A/B/C—C11A/B/C—O11A/B/C = 157.2 (4) and 174.8 (4)$^\circ$, respectively. In the torsion angles of the nitro groups C2A/B/C—C3A/B/C—N3A/B/C—O32A/B/C are $-150.4$ (12), 174.1 (16) and $120.3$ (13)$^\circ$, respectively. In the structure of the parent CLNBA acid (Ishida & Fukunaga, 2003), the corresponding torsion angles are 174.02 (17) and $-132.61$ (18)$^\circ$ compared to 179.7 (2) and $-137.8$ (2)$^\circ$ in the Na–CLNBA monohydrate salt (Smith, 2013).

### Table 2

| Hydrogen-bond geometry (Å, $^\circ$) for (I). |
|---------------------------------------------|
| $D$—H · · · $A$ | $D$—H | H · · · $A$ | $D$—$A$ | $D$—H · · · $A$ |
|-----------------|------|----------|-------|-----------------|
| O1W—H11W···O11A$^i$ | 0.82 (4) | 1.95 (4) | 2.757 (4) | 166 (4) |
| O1W—H12W···O11B$^ii$ | 0.82 (3) | 1.98 (3) | 2.777 (4) | 163 (4) |
| O2W—H21W···N4B$^iii$ | 0.84 (4) | 2.09 (4) | 2.902 (5) | 162 (5) |
| O2W—H22W···N4C$^iv$ | 0.86 (4) | 1.89 (4) | 2.735 (6) | 168 (5) |
| O3W—H31W···O12B | 0.83 (4) | 1.99 (4) | 2.777 (4) | 160 (5) |
| O3W—H32W···O12A$^v$ | 0.85 (3) | 2.07 (3) | 2.841 (5) | 151 (5) |
| N4A···H42A···O3B$^vi$ | 0.88 (4) | 2.08 (4) | 2.902 (6) | 156 (4) |
| N4B···H41B···O3W$^vii$ | 0.86 (4) | 2.18 (4) | 3.014 (6) | 164 (4) |
| N4C···H42C···O1B$^iii$ | 0.86 (3) | 2.49 (4) | 3.341 (5) | 170 (5) |

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $-x+2, -y+1, -z+1$; (iii) $x, y-1, z$; (iv) $x+1, y+1, z$; (v) $-x+1, -y+1, -z+2$; (vi) $-x, -y+1, -z+2$; (vii) $-x+1, -y+2, -z+1$.

### Table 3

| Selected bond lengths (Å) for (II). |
|-------------------------------------|
| $D$—H · · · $A$ | $D$—H | H · · · $A$ | $D$—$A$ | $D$—H · · · $A$ |
|-----------------|------|----------|-------|-----------------|
| Er1—O11 | 2.306 (7) | 2.312 (8) | 2.317 (7) | 2.313 (6) |
| Er1—O11C | 2.306 (7) | 2.312 (8) | 2.317 (7) | 2.313 (6) |
| Er1—O12B | 2.306 (7) | 2.312 (8) | 2.317 (7) | 2.313 (6) |
| Er1—O12C$^i$ | 2.287 (6) | 2.300 (6) | 2.348 (6) | 2.348 (6) |

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $-x+1, -y+1, -z+1$.

### Table 4

| Hydrogen-bond geometry (Å, $^\circ$) for (II). |
|---------------------------------------------|
| $D$—H · · · $A$ | $D$—H | H · · · $A$ | $D$—$A$ | $D$—H · · · $A$ |
|-----------------|------|----------|-------|-----------------|
| C2A···H2A···S1 | 0.95 | 2.86 | 3.743 (10) | 155 |
| C2B···H2B···O11 | 0.95 | 2.56 | 3.298 (15) | 135 |
| C11···H111···C14$^i$ | 0.98 | 2.79 | 3.486 (11) | 129 |
| C12···H123···O32A$^ii$ | 0.98 | 2.44 | 3.376 (15) | 158 |

Symmetry codes: (iii) $-x+1, -y+1, -z$; (iv) $-x, -y+1, -z$.

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**Figure 2**

The molecular configuration and atom-naming scheme for the centro-symmetric dinuclear repeat unit in the polymeric complex (II), with displacement ellipsoids drawn at the 40% probability level. [Symmetry code: (v) $x+1, y, z$; for other symmetry codes, see Table 3.]

**Figure 3**

The packing of the one-dimensional polymeric chain structure of (II) in the unit cell, viewed approximately along [001]. H atoms have been omitted.
3. Supramolecular features

In the crystal structure of compound (I), extensive inter-unit \(\text{O–H} \cdots \text{O}\) and \(\text{O–H} \cdots \text{N}\) hydrogen-bonding interactions are present, involving both the coordinating water molecules as well as the solvent water molecules, with carboxylate O-atom acceptors and amine N-atom acceptors (Table 2). These, together with amine N–H\(\cdots\)O\(_\text{water}\) and O\(\cdots\)carboxyl hydrogen bonds give a three-dimensional network structure (Figs. 4 and 5). One H atom of each of the amine groups on the three 4-ABA ligand components of the complex is not involved in hydrogen-bonding. Also present in the supramolecular structure are weak \(\text{C} \cdots \text{C}\) interactions between A ligands \([\text{ring-centroid separation A} \cdots \text{A}^\text{vii} = 3.711 (3) \text{Å}]\) and C ligands \([\text{C} \cdots \text{C}^\text{viii} = 3.676 (3) \text{Å}\] (for symmetry codes, see Table 2). This dimeric carboxylate-bridged complex mode is similar to that found in the erbium acetate complex \([\text{Er}_2(\text{CH}_3\text{CO}_2)_6(\text{H}_2\text{O})_4]^2\text{H}_2\text{O}\) (Sawase et al., 1984).

With (II), present are two weak intra-polymer C–H\(\cdots\)O hydrogen bonds involving methyl H atoms and both a DMSO O-atom acceptor and a Cl-atom acceptor (Table 4).

4. Synthesis and crystallization

The title compounds were synthesized by warming together for 10 min, a solution obtained by mixing 5 ml of ethanolic 4-aminobenzoic acid (1 mmol: 135 mg) [for (I)] or 4-chloro-3-nitrobenzoic acid (1 mmol: 200 mg) [for (II)], with 10 ml of aqueous erbium(III) acetate hexahydrate (0.3 mmol: 216 mg). Partial room-temperature evaporation of these solutions provided pale-pink block-like single crystals of (I), suitable for X-ray analysis while a colourless powder was obtained from the preparation of (II). Recrystallization using the slow diffusion of water into a DMSO solution gave minor small crystals of (II), suitable for X-ray analysis.

5. Refinement details

Crystal data, data collection and structure refinements for (I) and (II) are summarized in Table 5. Hydrogen atoms on all water molecules and the amine groups of the 4-ABA ligands in (I) were located by difference methods and positional parameters were refined with restraints \([\text{O–H} \text{bond length} = 0.85 (2) \text{Å} \text{and} \text{N–H} = 0.88 (2) \text{Å}]\) with \(U_{\text{iso}}(\text{H}) = 1.5 U_{eq}(\text{O})\) or \(1.2 U_{eq}(\text{N})\). Other H atoms were included in the refinement at calculated positions \([\text{C–H(aromatic)} = 0.95 \text{Å} \text{or} \text{C–H(methyl)} = 0.96 \text{Å}\] with \(U_{\text{iso}}(\text{H}) = 1.2 U_{eq}(\text{C})(\text{aromatic})\) or \(1.5 U_{eq}(\text{C})(\text{methyl})\] using a riding-model approximation. In the refinement of (II), a number of large difference electron density residual peaks \((5–7 \text{e Å}^{-3})\] located within 1.0 Å of the \(\text{Er}^1\) site were present. These are possibly due to poor crystal quality coupled to effects of an insufficient absorption correction.

Acknowledgements

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References

Agilent (2013). CrysAlis PRO. Agilent Technologies Ltd, Yarnton, England.
Altomare, A., Cascarano, G., Giacovazzo, C. & Guagliardi, A. (1993). J. Appl. Cryst. 26, 343–350.
Chadwick, K., Sadiq, G., Davey, R. J., Seaton, C. C., Pritchard, R. G. & Parkin, A. (2009). Cryst. Growth Des. 9, 1278–1279.
Farrugia, L. J. (2012). J. Appl. Cryst. 45, 849–854.
Gracin, S. & Fischer, A. (2005). Acta Cryst. E61, o1242–o1244.
Ishida, H. & Fukunaga, T. (2003). Acta Cryst. E59, o1984–o1986.
Sastry, V. S., Bünzli, J.-C., Ramachandra Rao, V., Rayudi, G. V. S. & Perumareddi, J. R. (2003). In Modern Aspects of Rare Earths and Their Complexes. Amsterdam: Elsevier.
Table 5
Experimental details.

|                | (I)                                                                 | (II)                                                                 |
|----------------|---------------------------------------------------------------------|---------------------------------------------------------------------|
| Crystal data   |                                                                     |                                                                     |
| Chemical formula | [Er₂(C₇H₆NO₂)₆(H₂O)₄]·2H₂O                                         | [Er₂(C₇H₃ClNO₄)₆(C₂H₆OS)₂]                                         |
| Mr             | 1259.38                                                            | 1694.10                                                            |
| Crystal system, space group | Triclinic, P₁                                            | Triclinic, P₁                                                        |
| Temperature (K) | 200                                                                | 200                                                                |
| a, b, c (Å)    | 9.0332 (5), 10.9363 (6), 12.6194 (6)                               | 8.2408 (3), 12.4040 (8), 15.3409 (10)                              |
| α, β, γ (°)    | 89.015 (4), 72.105 (5), 74.814 (5)                                 | 111.443 (6), 98.063 (4), 96.684 (4)                                |
| V (Å³)         | 1142.21 (10)                                                       | 1421.04 (14)                                                       |
| Z              | 1                                                                  | 1                                                                  |
| Radiation type | Mo Kα                                                               | Mo Kα                                                               |
| μ (mm⁻¹)       | 3.73                                                               | 3.38                                                               |
| Crystal size (mm) | 0.30 × 0.30 × 0.25                                                | 0.25 × 0.12 × 0.04                                                 |

Data collection

|                |                                                                  |                                                                  |
|----------------|-----------------------------------------------------------------|-----------------------------------------------------------------|
| Diffraetometer | Oxford Diffraction Gemini-S CCD detector                      | Oxford Diffraction Gemini-S CCD detector                      |
| Absorption correction | Multi-scan (CrysAlis PRO; Agilent, 2013)                  | Multi-scan (CrysAlis PRO; Agilent, 2013)                  |
| Tₘin, Tₘax     | 0.713, 0.980                                                     | 0.494, 0.980                                                     |
| No. of measured, independent and observed | 7274, 4480, 4137                                             | 10041, 5566, 4814                                             |
| F > 2σ(F)      | 0.035                                                           | 0.055                                                           |
| (sin θ/λ)max (Å⁻¹) | 0.617                                                         | 0.617                                                         |

Refinement

|                |                                                                 |                                                                 |
|----------------|-----------------------------------------------------------------|-----------------------------------------------------------------|
| R[F² > 2σ(F²)], wR(F²), S | 0.029, 0.058, 1.05                                              | 0.067, 0.181, 1.06                                              |
| No. of reflections | 4480                                                            | 5566                                                           |
| No. of parameters | 343                                                             | 397                                                            |
| No. of restraints | 12                                                              | 0                                                              |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement | H-atom parameters constrained                                  |
| Δρmax, Δρmin (e Å⁻³) | 1.03, −0.71                                                      | 6.83, −2.41                                                     |

Computer programs: CrysAlis PRO (Agilent, 2013), SIR92 (Altomare et al., 1993), SHELX97 and SHELXL97 (Sheldrick, 2008) within WinGX (Farrugia, 2012) and PLATON (Spek, 2009).

Sawase, H., Koizumi, Y., Suzuki, Y., Shimoi, M. & Ouchi, Z. (1984). Bull. Chem. Soc. Jpn., 57, 2730–2737.
Sheldrick, G. M. (2008). Acta Cryst. A64, 112–122.
Smith, G. (2013). Acta Cryst. C69, 1472–1477.
Speck, A. L. (2009). Acta Cryst. D65, 148–155.
Sun, H.-L., Ye, C.-H., Wang, X.-Y., Li, J.-R., Gao, S. & Yu, K.-B. (2004). J. Mol. Struct. 702, 77–83.
Crystal structures of two erbium(III) complexes with 4-aminobenzoic acid and 4-chloro-3-nitrobenzoic acid

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Computing details
For both compounds, data collection: CrysAlis PRO (Agilent, 2013); cell refinement: CrysAlis PRO (Agilent, 2013); data reduction: CrysAlis PRO (Agilent, 2013). Program(s) used to solve structure: SIR92 (Altomare et al., 1993) for (I); SHELXS97 (Sheldrick, 2008) for (II). For both compounds, program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) within WinGX (Farrugia, 2012); molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: PLATON (Spek, 2009).

(I) Bis(μ₂-4-aminobenzoato-κ²O,O')bis(4-aminobenzoato-κ²O,O')diaquaerbium(III) dihydrate

Crystal data
\[\text{Er}_2(C_7H_6NO_2)_6(H_2O)_4\cdot2H_2O\]
\[M_r = 1259.38\]
Triclinic, \(P\bar{1}\)
Hall symbol: -P 1
\(a = 9.0332\) (5) Å
\(b = 10.9363\) (6) Å
\(c = 12.6194\) (6) Å
\(\alpha = 89.015\) (4)°
\(\beta = 72.105\) (5)°
\(\gamma = 74.814\) (5)°
\(V = 1142.21\) (10) Å³

Data collection
Oxford Diffraction Gemini-S CCD-detector diffractometer
Radiation source: Enhance (Mo) X-ray source
Graphite monochromator
Detector resolution: 16.077 pixels mm⁻¹
\(\omega\) scans
Absorption correction: multi-scan
(CrysAlis PRO; Agilent, 2013)
\(T_{\text{min}} = 0.713, T_{\text{max}} = 0.980\)

Refinement
Refinement on \(F^2\)
Least-squares matrix: full
\(R[F^2 > 2\sigma(F^2)] = 0.029\)
\(wR(F^2) = 0.058\)
\(S = 1.05\)
4480 reflections
343 parameters
12 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement

\[ w = \frac{1}{\sigma^2(F_o^2) + (0.011P)^2} \]
where \( P = (F_o^2 + 2F_c^2)/3 \)

\( (\Delta/\sigma)_{\text{max}} = 0.002 \)
\( \Delta \rho_{\text{max}} = 1.03 \, \text{e} \, \text{Å}^{-3} \)
\( \Delta \rho_{\text{min}} = -0.71 \, \text{e} \, \text{Å}^{-3} \)

**Special details**

**Geometry.** Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su’s are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.’s are taken into account in the estimation of distances, angles and torsion angles

**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   | \( U_{eq} \)/\( U_{eq} \) |
|----|-------|-------|-------|--------------------------|
| Er1| 0.63868 (2) | 0.48651 (2) | 0.63400 (1) | 0.0172 (1) |
| O1W| 0.8216 (3)  | 0.4689 (3)  | 0.4504 (2)  | 0.0236 (9)  |
| O2W| 0.8310 (4)  | 0.3257 (3)  | 0.6738 (3)  | 0.0310 (10) |
| O3W| 0.4420 (4)  | 0.6614 (4)  | 1.0062 (3)  | 0.0450 (13) |
| O11A| 0.3424 (3) | 0.5182 (3) | 0.7029 (2) | 0.0229 (9) |
| O11B| 0.8438 (3) | 0.5973 (3) | 0.6381 (2) | 0.0226 (9) |
| O11C| 0.4044 (4) | 0.6642 (3) | 0.4607 (3) | 0.0393 (11) |
| O12A| 0.4967 (3) | 0.3977 (3) | 0.7885 (2) | 0.0279 (10) |
| O12B| 0.6239 (4) | 0.6268 (3) | 0.7818 (2) | 0.0297 (10) |
| O12C| 0.5398 (3) | 0.6760 (3) | 0.5771 (2) | 0.0326 (10) |
| N4A| −0.1513 (5) | 0.2669 (5) | 1.0592 (3) | 0.0420 (16) |
| N4B| 0.8254 (5)  | 1.1355 (4)  | 0.8371 (3)  | 0.0338 (14) |
| N4C| 0.1613 (5)  | 1.2581 (4)  | 0.5966 (4)  | 0.0408 (14) |
| C1A| 0.2234 (5)  | 0.3912 (4)  | 0.8436 (3)  | 0.0205 (12) |
| C1B| 0.7719 (5)  | 0.7812 (4)  | 0.7614 (3)  | 0.0209 (11) |
| C1C| 0.3588 (4)  | 0.8633 (4)  | 0.5499 (3)  | 0.0173 (11) |
| C2A| 0.2533 (5)  | 0.2916 (4)  | 0.9109 (3)  | 0.0259 (12) |
| C2B| 0.6425 (5)  | 0.8743 (4)  | 0.8302 (3)  | 0.0245 (12) |
| C2C| 0.3840 (5)  | 0.9328 (4)  | 0.6308 (3)  | 0.0239 (12) |
| C3A| 0.1314 (5)  | 0.2489 (4)  | 0.9799 (3)  | 0.0286 (16) |
| C3B| 0.6601 (5)  | 0.9903 (4)  | 0.8557 (3)  | 0.0269 (12) |
| C3C| 0.3173 (5)  | 1.0619 (4)  | 0.6478 (3)  | 0.0297 (14) |
| C4A| −0.0285 (5) | 0.3068 (4)  | 0.9855 (3)  | 0.0272 (16) |
| C4B| 0.8090 (5)  | 1.0165 (4)  | 0.8158 (3)  | 0.0238 (14) |
| C4C| 0.2265 (5)  | 1.1253 (4)  | 0.5836 (4)  | 0.0264 (14) |
| C5A| −0.0601 (5) | 0.4036 (4)  | 0.9147 (3)  | 0.0284 (14) |
| C5B| 0.9399 (5)  | 0.9232 (4)  | 0.7501 (3)  | 0.0263 (12) |
| C5C| 0.1958 (5)  | 1.0556 (4)  | 0.5055 (3)  | 0.0295 (14) |
| C6A| 0.0656 (5)  | 0.4452 (4)  | 0.8453 (3)  | 0.0240 (12) |
| C6B| 0.9214 (5)  | 0.8076 (4)  | 0.7221 (3)  | 0.0243 (12) |
| C6C| 0.2620 (5)  | 0.9257 (4)  | 0.4890 (3)  | 0.0272 (14) |
### Atomic displacement parameters (Å²)

| Atom  | U¹¹  | U¹²  | U¹³  | U¹²  | U¹³  | U¹³  |
|-------|------|------|------|------|------|------|
| Er1   | 0.0190 (1) | 0.0146 (1) | 0.0170 (1) | -0.0037 (1) | -0.0048 (1) | 0.0002 (1) |
| O1W   | 0.0257 (16) | 0.0120 (17) | 0.0175 (15) | -0.0052 (15) | -0.0073 (15) | 0.0014 (15) |
| O2W   | 0.0154 (16) | 0.0156 (15) | 0.0141 (14) | -0.0052 (14) | -0.0063 (14) | 0.0006 (14) |
| O11A  | 0.0220 (15) | 0.0180 (15) | 0.0201 (14) | -0.0065 (13) | -0.0076 (13) | 0.0010 (13) |
| O11B  | 0.0244 (16) | 0.0164 (15) | 0.0196 (15) | -0.0058 (15) | -0.0070 (15) | 0.0013 (15) |
| O12B  | 0.0344 (12) | 0.0238 (12) | 0.0230 (12) | -0.0134 (12) | -0.0155 (12) | 0.0040 (12) |
| N4A   | 0.025 (3) | 0.014 (3) | 0.014 (3) | -0.003 (3) | -0.010 (3) | 0.002 (3) |
| N4B   | 0.027 (3) | 0.022 (2) | 0.020 (2) | -0.003 (2) | -0.009 (2) | 0.001 (2) |
| C1A   | 0.024 (2) | 0.020 (2) | 0.017 (2) | -0.006 (2) | -0.007 (2) | 0.002 (2) |
| C1B   | 0.028 (2) | 0.021 (2) | 0.013 (2) | -0.004 (2) | -0.006 (2) | 0.001 (2) |
| C1C   | 0.0153 (19) | 0.016 (2) | 0.019 (2) | -0.0049 (16) | -0.0023 (16) | 0.0001 (16) |
| C2A   | 0.024 (2) | 0.028 (2) | 0.028 (2) | -0.0071 (19) | -0.0114 (19) | 0.0045 (19) |
| C2B   | 0.025 (2) | 0.025 (2) | 0.021 (2) | -0.0074 (19) | -0.0033 (18) | 0.0024 (18) |
Geometric parameters (Å, º)

Er1—O1W 2.373 (2)  C1A—C2A 1.391 (6)
Er1—O2W 2.295 (3)  C1B—C2B 1.393 (6)
Er1—O11A 2.477 (3)  C1B—C6B 1.393 (7)
Er1—O11B 2.478 (3)  C1B—C11B 1.480 (6)
Er1—O12A 2.333 (3)  C1C—C11C 1.490 (6)
Er1—O12B 2.385 (3)  C1C—C6C 1.380 (6)
Er1—O12C 2.232 (3)  C1C—C2C 1.390 (6)
Er1—O11Ci 2.233 (4)  C2A—C3A 1.362 (6)
O11A—C11A 1.257 (5)  C2B—C3B 1.375 (6)
O11B—C11B 1.262 (5)  C2B—C6B 1.374 (6)
O11C—C11C 1.245 (6)  C3A—C4A 1.397 (7)
O12A—C11A 1.273 (6)  C3B—C4B 1.388 (7)
O12B—C11B 1.273 (6)  C3C—C4C 1.379 (6)
O12C—C11C 1.254 (5)  C4A—C5A 1.402 (6)
O1W—H12W 0.82 (3)  C4B—C5B 1.386 (6)
O1W—H11W 0.82 (4)  C4C—C5C 1.391 (6)
O2W—H21W 0.84 (4)  C5A—C6A 1.382 (6)
O2W—H22W 0.86 (4)  C5B—C6B 1.383 (6)
O3W—H31W 0.83 (4)  C5C—C6C 1.381 (6)
O3W—H32W 0.85 (5)  C2A—H2A 0.9300
N4A—C4A 1.375 (6)  C2B—H2B 0.9300
N4B—C4B 1.388 (6)  C2C—H2C 0.9300
N4C—C4C 1.409 (6)  C3A—H3A 0.9300
N4A—H41A 0.87 (4)  C3B—H3B 0.9300
N4A—H42A 0.88 (4)  C3C—H3C 0.9300
N4B—H41B 0.86 (4)  C5A—H5A 0.9300
N4B—H42B 0.86 (3)  C5B—H5B 0.9300
N4C—H41C 0.89 (5)  C5C—H5C 0.9300

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| Bond                      | Distance (Å) | Bond                      | Distance (Å) | Distance (Å) |
|---------------------------|--------------|---------------------------|--------------|--------------|
| N4C—H42C                 | 0.86 (3)     | C6A—H6A                  | 0.9300       |
| C1A—C11A                 | 1.482 (6)    | C6B—H6B                  | 0.9300       |
| C1A—C6A                  | 1.386 (7)    | C6C—H6C                  | 0.9300       |
| O1W—Er1—O2W              | 87.02 (12)   | C2C—C1C—C6C              | 118.8 (4)    |
| O1W—Er1—O11A             | 131.43 (9)   | C6C—C1C—C11C             | 121.1 (4)    |
| O1W—Er1—O11B             | 72.20 (9)    | C1A—C2A—C3A              | 121.6 (4)    |
| O1W—Er1—O12A             | 151.62 (11)  | C1B—C2B—C3B              | 121.2 (4)    |
| O1W—Er1—O12B             | 124.33 (11)  | C1C—C2C—C3C              | 120.6 (4)    |
| O1W—Er1—O12C             | 79.98 (10)   | C1C—C2C—C3C              | 120.6 (4)    |
| O1W—Er1—O11C             | 73.68 (12)   | C2B—C3B—C4B              | 120.7 (4)    |
| O2W—Er1—O11A             | 126.78 (12)  | C2C—C3C—C4C              | 120.6 (4)    |
| O2W—Er1—O11B             | 78.50 (12)   | C3A—C4A—C5A              | 119.1 (4)    |
| O2W—Er1—O12A             | 75.02 (12)   | N4A—C4A—C5A              | 121.4 (4)    |
| O2W—Er1—O12B             | 93.16 (12)   | N4A—C4A—C5A              | 119.5 (4)    |
| O2W—Er1—O12C             | 156.11 (12)  | C3B—C4B—C5B              | 118.7 (4)    |
| O2W—Er1—O11C             | 85.80 (13)   | C4B—C5B—C6B              | 120.5 (4)    |
| O11A—Er1—O11B            | 140.04 (10)  | N4B—C4B—C3B              | 120.8 (4)    |
| O11A—Er1—O12A            | 53.86 (10)   | C3C—C4C—C5C              | 118.9 (4)    |
| O11A—Er1—O12B            | 91.09 (11)   | N4C—C4C—C5C              | 121.9 (4)    |
| O11A—Er1—O12C            | 76.09 (10)   | N4C—C4C—C5C              | 119.2 (4)    |
| O11A—Er1—O11C            | 75.35 (12)   | C4A—C5A—C6A              | 119.8 (4)    |
| O11B—Er1—O12A            | 123.63 (9)   | C4B—C5B—C6B              | 120.6 (4)    |
| O11B—Er1—O12B            | 53.56 (10)   | C4C—C5C—C6C              | 120.3 (4)    |
| O11B—Er1—O12C            | 78.48 (10)   | C1A—C6A—C5A              | 120.8 (4)    |
| O11B—Er1—O11C            | 142.95 (11)  | C1B—C6B—C5B              | 120.8 (4)    |
| O12A—Er1—O12B            | 79.21 (10)   | C1C—C6C—C5C              | 120.6 (4)    |
| O12A—Er1—O12C            | 123.94 (10)  | O11A—C11A—C1A            | 122.2 (4)    |
| O11C—Er1—O12A            | 83.11 (12)   | O12A—C11A—C1A            | 118.5 (4)    |
| O12B—Er1—O12C            | 78.15 (10)   | O11A—C11A—O12A           | 119.2 (4)    |
| O11C—Er1—O12B            | 161.93 (12)  | O11B—C11B—C1B            | 120.7 (4)    |
| O11C—Er1—O12C            | 109.26 (11)  | O12B—C11B—C1B            | 119.4 (3)    |
| Er1—O11A—C11A            | 90.0 (3)     | O11B—C11B—O12B           | 119.8 (4)    |
| Er1—O11B—C11B            | 90.2 (3)     | O11C—C11C—O12C           | 124.0 (4)    |
| Er1—O11C—C11C            | 165.0 (3)    | O11C—C11C—C1C            | 117.9 (4)    |
| Er1—O12A—C11A            | 96.3 (2)     | O12C—C11C—C1C            | 118.1 (4)    |
| Er1—O12B—C11B            | 94.2 (2)     | C1A—C2A—H2A              | 119.00       |
| Er1—O12C—C11C            | 138.1 (3)    | C3A—C2A—H2A              | 119.00       |
| H11W—O1W—H12W            | 100 (4)      | C3B—C2B—H2B              | 119.00       |
| Er1—O1W—H11W             | 119 (3)      | C1B—C2B—H2B              | 119.00       |
| Er1—O1W—H12W             | 141 (2)      | C1C—C2C—H2C              | 120.00       |
| H21W—O2W—H22W            | 107 (5)      | C3C—C2C—H2C              | 120.00       |
| Er1—O2W—H21W             | 122 (4)      | C4A—C3A—H3A              | 120.00       |
| Er1—O2W—H22W             | 130 (3)      | C2A—C3A—H3A              | 120.00       |
| H31W—O3W—H32W            | 104 (5)      | C2B—C3B—H3B              | 120.00       |
| C4A—N4A—H41A             | 121 (4)      | C4B—C3B—H3B              | 120.00       |
| H41A—N4A—H42A            | 122 (5)      | C4C—C3C—H3C              | 120.00       |
| C4A—N4A—H42A             | 115 (3)      | C2C—C3C—H3C              | 120.00       |
C4B—N4B—H42B 111 (4)    C4A—C5A—H5A 120.00
H41B—N4B—H42B 112 (4)    C6A—C5A—H5A 120.00
C4B—N4B—H41B 116 (3)     C6B—C5B—H5B 120.00
C4C—N4C—H41C 108 (3)     C4B—C5B—H5B 120.00
H41C—N4C—H42C 121 (5)    C4C—C5C—H5C 120.00
C4C—N4C—H42C 110 (3)     C6C—C5C—H5C 120.00
C2A—C1A—C6A 118.6 (4)    C5A—C6A—H6A 120.00
C6A—C1A—C11A 121.7 (4)   C1A—C6A—H6A 120.00
C2A—C1A—C11A 119.7 (4)   C2B—C1B—C6B 120.00
C2B—C1B—C11B 120.6 (4)  C5B—C6B—H6B 120.00
C6B—C1B—C11B 121.3 (4)  C1C—C6C—H6C 120.00
C2B—C1B—C6B 118.0 (4)   C5C—C6C—H6C 120.00
C2C—C1C—C11C 120.1 (4)  O1W—Er1—O11A—C11A 139.1 (2)  Er1—O12C—C11C—C1C 153.5 (3)
O2W—Er1—O11A—C11A 14.1 (3)  C2A—C1A—C6A—C5A 1.9 (6)
O11B—Er1—O11A—C11A −106.2 (3)  C6A—C1A—C2A—C3A −1.8 (6)
O12A—Er1—O11A—C11A −4.9 (2)  C11A—C1A—C2A—C3A 176.4 (4)
O12B—Er1—O11A—C11A −80.7 (2)  C6A—C1A—C11A—O11A −10.6 (6)
O12C—Er1—O11A—C11A −158.2 (2)  C6A—C1A—C11A—O12A 170.5 (4)
O11C—Er1—O11A—C11A 87.3 (2)  C1A—C6A—C11A—O11A −10.6 (6)
O1W—Er1—O11B—C11B 158.2 (3)  C2A—C1A—C11A—O12A 171.2 (4)
O2W—Er1—O11B—C11B −111.2 (2)  C2A—C1A—C11A—O11A −7.7 (6)
O11A—Er1—O11B—C11B 23.9 (3)  C6B—C1B—C2B—C3B 2.1 (6)
O12A—Er1—O11B—C11B −48.1 (3)  C11B—C1B—C2B—C3B −174.4 (4)
O12B—Er1—O11B—C11B −8.5 (2)  C2B—C1B—C6B—C5B −0.5 (6)
O12C—Er1—O11B—C11B 75.2 (2)  C2B—C1B—C11B—O11B 155.9 (4)
O11C—Er1—O11B—C11B −178.1 (2)  C2B—C1B—C11B—O12B −19.8 (6)
O1W—Er1—O12A—C11A −107.0 (3)  C6B—C1B—C11B—O11B −20.5 (6)
O2W—Er1—O12A—C11A −159.5 (3)  C6B—C1B—C11B—O12B 163.9 (4)
O11A—Er1—O12A—C11A 4.9 (2)  C11B—C1B—C6B—C5B 176.0 (4)
O11B—Er1—O12A—C11A 135.7 (2)  C6C—C1C—C2C—C3C −1.7 (6)
O12B—Er1—O12A—C11A 104.2 (3)  C11C—C1C—C2C—C3C 176.5 (4)
O12C—Er1—O12A—C11A 36.6 (3)  C2C—C1C—C11C—O12C −5.8 (6)
O11C—Er1—O12A—C11A −72.0 (2)  C6C—C1C—C11C—O11C −7.1 (6)
O1W—Er1—O12B—C11B −6.9 (3)  C6C—C1C—C11C—O12C 172.3 (4)
O2W—Er1—O12B—C11B 81.7 (3)  C2C—C1C—C6C—C5C 2.2 (6)
O11A—Er1—O12B—C11B −151.4 (3)  C11C—C1C—C6C—C5C −176.0 (4)
O11B—Er1—O12B—C11B 8.5 (2)  C2C—C1C—C11C—O11C 174.8 (4)
O12A—Er1—O12B—C11B 155.8 (3)  C1A—C2A—C3A—C4A −1.0 (6)
O12C—Er1—O12B—C11B −75.9 (3)  C1B—C2B—C3B—C4B −1.8 (6)
O1W—Er1—O12C—C11C 88.7 (4)  C1C—C2C—C3C—C4C −1.2 (7)
O2W—Er1—O12C—C11C 146.8 (4)  C2A—C3A—C4A—C5A 3.7 (6)
O11A—Er1—O12C—C11C −48.7 (4)  C2A—C3A—C4A—N4A −177.0 (4)
O11B—Er1—O12C—C11C 162.4 (4)  C2B—C3B—C4B—C5B −0.3 (6)
O12A—Er1—O12C—C11C −74.6 (4)  C2B—C3B—C4B—N4B 177.0 (4)
O12B—Er1—O12C—C11C −142.8 (4)  C2C—C3C—C4C—N4C −177.8 (4)
O11C—Er1—O12C—C11C 20.1 (4)  C2C—C3C—C4C—C5C 3.6 (7)

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Er1—O11A—C11A—O12A 8.3 (4) N4A—C4A—C5A—C6A 177.1 (4)
Er1—O11A—C11A—C1A −170.6 (3) C3A—C4A—C5A—C6A −3.7 (6)
Er1—O11B—C11B—O12B 14.9 (4) C3B—C4B—C5B—C6B 1.9 (6)
Er1—O11B—C11B—C1B −160.7 (4) N4B—C4B—C5B—C6B −175.4 (4)
Er1—O12A—C11A—O11A −8.9 (4) C3C—C4C—C5C—C6C −3.1 (7)
Er1—O12A—C11A—C1A 170.1 (3) N4C—C4C—C5C—C6C 178.2 (4)
Er1—O12B—C11B—O11B −15.5 (4) C4A—C5A—C6A—C1A 0.9 (6)
Er1—O12B—C11B—C1B 160.1 (3) C4B—C5B—C6B—C1B −1.5 (6)
Er1—O12C—C11C—O11C −27.1 (6) C4C—C5C—C6C—C1C 0.2 (7)

Symmetry code: (i) −x+1, −y+1, −z+1.

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|------|------|---------|
| O1W—H11W···O11A | 0.82 (4) | 1.95 (4) | 2.757 (4) | 166 (4) |
| O1W—H12W···O11B | 0.82 (3) | 1.98 (3) | 2.777 (4) | 163 (4) |
| O2W—H21W···N4B | 0.84 (4) | 2.09 (4) | 2.902 (5) | 162 (5) |
| O2W—H22W···N4C | 0.86 (4) | 1.89 (4) | 2.735 (6) | 168 (5) |
| O3W—H31W···O12B | 0.83 (4) | 1.99 (4) | 2.777 (4) | 160 (5) |
| O3W—H32W···O12A | 0.85 (5) | 2.07 (5) | 2.841 (5) | 151 (5) |
| N4A—H41A···O3W | 0.86 (4) | 2.08 (4) | 2.902 (6) | 156 (5) |
| N4A—H42A···O3W | 0.86 (3) | 2.49 (4) | 3.341 (5) | 170 (5) |

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

(II) Poly[hexakis(μ-2-4-chloro-3-nitrobenzoato-κ<sup>2</sup>O<sup>2</sup>:O′)bis(dimethyl sulfoxide-κO)dierbium(III)]

Crystal data

[Er<sub>2</sub>(C<sub>7</sub>H<sub>3</sub>ClNO<sub>4</sub>)<sub>6</sub>(C<sub>2</sub>H<sub>6</sub>OS)<sub>2</sub>]

Mr = 1694.10

Triclinic, P<sup>1</sup>

Hall symbol: -P 1

a = 8.2408 (3) Å

b = 12.4040 (8) Å

c = 15.3409 (10) Å

α = 111.443 (6)°

β = 98.063 (4)°

γ = 96.684 (4)°

V = 1421.04 (14) Å<sup>3</sup>

Data collection

Oxford Diffraction Gemini-S CCD-detector

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 16.077 pixels mm<sup>−1</sup>

0 scans

Absorption correction: multi-scan

(Crystalis PRO; Agilent, 2013)

T<sub>min</sub> = 0.494, T<sub>max</sub> = 0.980
**Refinement**

Refinement on $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.067$

$wR(F^2) = 0.181$

$S = 1.06$

5566 reflections

397 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/\left[\sigma^2(F_o^2) + (0.1243P)^2\right]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 6.83 \text{ e Å}^{-3}$

$\Delta\rho_{\text{min}} = -2.41 \text{ e Å}^{-3}$

**Special details**

*Geometry.* Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**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 ($\text{Å}^2$)**

|   | $x$     | $y$     | $z$     | $U_{eq}$/$U_{eq}$ |
|---|---------|---------|---------|-------------------|
|Er1| 0.24949 (4) | 0.48443 (3) | 0.46092 (2) | 0.0175 (1) |
|Cl4A| 0.6408 (5) | 0.7116 (4) | 0.0335 (3) | 0.0699 (16) |
|Cl4B| 0.2887 (4) | −0.1334 (3) | 0.0200 (2) | 0.0627 (10) |
|Cl4C| −0.3399 (4) | −0.1283 (2) | 0.5158 (2) | 0.0452 (9) |
|S | 0.0342 (3) | 0.4386 (2) | 0.23184 (16) | 0.0269 (7) |
|O11| 0.1349 (8) | 0.3972 (6) | 0.2999 (5) | 0.0294 (19) |
|O11A| 0.6659 (7) | 0.5633 (6) | 0.4105 (4) | 0.0252 (19) |
|O11B| 0.6883 (7) | 0.3352 (5) | 0.4066 (4) | 0.0254 (17) |
|O11C| 0.0768 (7) | 0.3102 (6) | 0.4347 (5) | 0.027 (2) |
|O12A| 0.3978 (7) | 0.5899 (6) | 0.3912 (4) | 0.0259 (17) |
|O12B| 0.4342 (7) | 0.3679 (5) | 0.4117 (4) | 0.0239 (17) |
|O12C| −0.0361 (7) | 0.4170 (5) | 0.5538 (5) | 0.0231 (19) |
|O31A| 0.1634 (13) | 0.6185 (12) | 0.0929 (8) | 0.079 (5) |
|O31B| −0.0284 (11) | 0.0741 (13) | 0.1852 (10) | 0.128 (6) |
|O31C| −0.1757 (16) | 0.1537 (14) | 0.7463 (8) | 0.112 (6) |
|O32A| 0.3085 (15) | 0.5798 (10) | −0.0175 (7) | 0.075 (4) |
|O32B| −0.0018 (15) | −0.0583 (16) | 0.0725 (12) | 0.174 (7) |
|O32C| −0.4244 (12) | 0.0843 (11) | 0.6745 (8) | 0.074 (4) |
|N3A| 0.2942 (15) | 0.6108 (9) | 0.0664 (7) | 0.050 (4) |
|N3B| 0.0575 (12) | 0.0190 (9) | 0.1417 (8) | 0.050 (3) |
|N3C| −0.2816 (13) | 0.1149 (8) | 0.6759 (7) | 0.043 (3) |
|C1A| 0.5617 (11) | 0.6222 (8) | 0.2856 (6) | 0.023 (2) |
|C1B| 0.4672 (11) | 0.1949 (8) | 0.2879 (6) | 0.023 (3) |
|C1C| −0.0974 (10) | 0.2075 (8) | 0.5005 (6) | 0.023 (3) |
|C2A| 0.4248 (11) | 0.6144 (8) | 0.2190 (6) | 0.025 (3) |
|C2B| 0.2996 (11) | 0.1571 (9) | 0.2529 (7) | 0.029 (3) |
| Atomic displacement parameters (Å²) | \( U_{11} \) | \( U_{22} \) | \( U_{33} \) | \( U_{12} \) | \( U_{13} \) | \( U_{23} \) |
|-----------------------------------|---------------|---------------|---------------|---------------|---------------|---------------|
| Er1                               | 0.0131 (2)    | 0.0219 (2)    | 0.0184 (2)    | 0.0064 (2)    | 0.0031 (2)    | 0.0079 (2)    |
| Cl4A                              | 0.092 (3)     | 0.105 (3)     | 0.061 (2)     | 0.055 (2)     | 0.052 (2)     | 0.063 (2)     |
| Cl4B                              | 0.0548 (18)   | 0.0484 (17)   | 0.0474 (18)   | 0.0099 (14)   | −0.0065 (14)  | −0.0183 (14)  |
| Cl4C                              | 0.0525 (17)   | 0.0285 (13)   | 0.0578 (18)   | −0.0038 (12)  | 0.0126 (13)   | 0.0231 (12)   |
| S1                                | 0.0227 (11)   | 0.0357 (13)   | 0.0211 (11)   | 0.0059 (9)    | 0.0017 (8)    | 0.0105 (9)    |
| O11                               | 0.031 (3)     | 0.031 (3)     | 0.026 (4)     | 0.016 (3)     | 0.002 (3)     | 0.009 (3)     |
| O11A                              | 0.017 (3)     | 0.035 (4)     | 0.025 (3)     | 0.010 (3)     | 0.005 (2)     | 0.012 (3)     |
| O11B                              | 0.016 (3)     | 0.033 (3)     | 0.028 (3)     | 0.005 (3)     | 0.004 (2)     | 0.012 (3)     |
| O11C                              | 0.016 (3)     | 0.035 (4)     | 0.028 (4)     | 0.000 (3)     | 0.005 (3)     | 0.012 (3)     |
| O12A                              | 0.021 (3)     | 0.035 (3)     | 0.028 (3)     | 0.008 (3)     | 0.006 (3)     | 0.018 (3)     |
| O12B                              | 0.020 (3)     | 0.026 (3)     | 0.027 (3)     | 0.010 (3)     | 0.008 (2)     | 0.009 (3)     |
| Atom   | U1     | U2     | U3     | U12    | U13    | U23    |
|--------|--------|--------|--------|--------|--------|--------|
| O12C   | 0.015  | 0.017  | 0.034  | 0.005  | 0.001  | 0.007  |
| O31A   | 0.048  | 0.135  | 0.064  | 0.021  | -0.010 | 0.057  |
| O31B   | 0.017  | 0.158  | 0.111  | -0.001 | 0.005  | -0.052 |
| O31C   | 0.094  | 0.168  | 0.041  | -0.061 | -0.002 | 0.035  |
| O32A   | 0.106  | 0.081  | 0.034  | 0.026  | -0.011 | 0.025  |
| O32B   | 0.039  | 0.176  | 0.154  | 0.001  | -0.016 | -0.092 |
| O32C   | 0.051  | 0.119  | 0.081  | 0.017  | 0.039  | 0.064  |
| N3A    | 0.060  | 0.058  | 0.033  | 0.020  | -0.009 | 0.022  |
| N3B    | 0.032  | 0.055  | 0.046  | 0.008  | -0.006 | 0.004  |
| N3C    | 0.059  | 0.035  | 0.034  | -0.001 | 0.015  | 0.014  |
| C1A    | 0.022  | 0.027  | 0.020  | 0.005  | 0.005  | 0.010  |
| C1B    | 0.017  | 0.027  | 0.023  | 0.005  | 0.002  | 0.007  |
| C1C    | 0.017  | 0.026  | 0.025  | 0.004  | 0.005  | 0.010  |
| C2A    | 0.021  | 0.032  | 0.020  | 0.006  | 0.002  | 0.009  |
| C2B    | 0.022  | 0.033  | 0.027  | 0.008  | 0.000  | 0.007  |
| C2C    | 0.015  | 0.028  | 0.030  | 0.001  | 0.003  | 0.015  |
| C3A    | 0.044  | 0.036  | 0.028  | 0.012  | 0.000  | 0.014  |
| C3B    | 0.022  | 0.032  | 0.035  | -0.003 | 0.001  | 0.007  |
| C3C    | 0.021  | 0.047  | 0.031  | 0.013  | 0.010  | 0.020  |
| C4A    | 0.050  | 0.047  | 0.030  | 0.018  | 0.021  | 0.023  |
| C4B    | 0.038  | 0.034  | 0.030  | 0.003  | 0.006  | 0.007  |
| C4C    | 0.025  | 0.023  | 0.042  | -0.004 | 0.001  | 0.022  |
| C5A    | 0.031  | 0.050  | 0.046  | 0.014  | 0.020  | 0.027  |
| C5B    | 0.033  | 0.034  | 0.036  | 0.013  | 0.010  | 0.004  |
| C5C    | 0.041  | 0.020  | 0.041  | 0.008  | 0.005  | 0.014  |
| C6A    | 0.015  | 0.045  | 0.042  | 0.007  | 0.007  | 0.018  |
| C6B    | 0.027  | 0.031  | 0.026  | 0.012  | 0.009  | 0.008  |
| C6C    | 0.029  | 0.019  | 0.031  | 0.002  | 0.010  | 0.011  |
| C11    | 0.028  | 0.069  | 0.023  | 0.014  | 0.007  | 0.014  |
| C11A   | 0.016  | 0.024  | 0.016  | 0.001  | 0.005  | 0.009  |
| C11B   | 0.010  | 0.021  | 0.027  | 0.007  | 0.004  | 0.012  |
| C11C   | 0.008  | 0.029  | 0.028  | 0.006  | -0.002 | 0.016  |
| C12    | 0.021  | 0.048  | 0.034  | 0.002  | 0.006  | 0.014  |

**Geometric parameters (Å, °)**

| Bond                  | Length (Å) | Angle (°) |
|-----------------------|------------|-----------|
| Er1—O11               | 2.306 (7)  | 1.419 (14)|
| Er1—O11C              | 2.312 (8)  | 1.496 (13)|
| Er1—O12A              | 2.317 (7)  | 1.398 (13)|
| Er1—O12B              | 2.239 (6)  | 1.387 (14)|
| Er1—O12C              | 2.287 (6)  | 1.507 (14)|
| Er1—O11A              | 2.300 (6)  | 1.386 (14)|
| Er1—O11B              | 2.348 (6)  | 1.390 (15)|
| Cl4A—C4A              | 1.729 (13) | 1.354 (16)|
| Cl4B—C4B              | 1.714 (11) | 1.361 (17)|
| Cl4C—C4C              | 1.730 (11) | 1.383 (15)|
| S1—O11                | 1.514 (8)  | 1.391 (15)|
| S1—C11                | 1.785 (10) | 1.396 (16)|

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| Bond               | Distance (Å) | Bond               | Distance (Å) | Bond               | Distance (Å) |
|--------------------|--------------|--------------------|--------------|--------------------|--------------|
| S1—C12             | 1.772 (11)   | C4B—C5B            | 1.391 (15)   |                   |              |
| O11A—C11A          | 1.274 (11)   | C4C—C5C            | 1.367 (15)   |                   |              |
| O11B—C11B          | 1.255 (10)   | C5A—C6A            | 1.368 (16)   |                   |              |
| O11C—C11C          | 1.255 (11)   | C5B—C6B            | 1.394 (15)   |                   |              |
| O12A—C11A          | 1.250 (10)   | C5C—C6C            | 1.391 (15)   |                   |              |
| O12B—C11B          | 1.249 (10)   | C2A—H2A            | 0.9500       |                   |              |
| O12C—C11C          | 1.271 (12)   | C2B—H2B            | 0.9500       |                   |              |
| O31A—N3A           | 1.206 (17)   | C2C—H2C            | 0.9500       |                   |              |
| O31B—N3B           | 1.151 (16)   | C5A—H5A            | 0.9500       |                   |              |
| O31C—N3C           | 1.191 (16)   | C5B—H5B            | 0.9500       |                   |              |
| O32A—N3A           | 1.229 (14)   | C5C—H5C            | 0.9500       |                   |              |
| O32B—N3B           | 1.13 (2)     | C6A—H6A            | 0.9500       |                   |              |
| O32C—N3C           | 1.188 (15)   | C6B—H6B            | 0.9500       |                   |              |
| N3A—C3A            | 1.480 (16)   | C6C—H6C            | 0.9500       |                   |              |
| N3B—C3B            | 1.474 (14)   | C11—H111           | 0.9800       |                   |              |
| N3C—C3C            | 1.481 (14)   | C11—H112           | 0.9800       |                   |              |
| C1A—C2A            | 1.380 (13)   | C11—H113           | 0.9800       |                   |              |
| C1A—C6A            | 1.386 (14)   | C12—H121           | 0.9800       |                   |              |
| C1A—C11A           | 1.524 (13)   | C12—H122           | 0.9800       |                   |              |
| C1B—C2B            | 1.369 (13)   | C12—H123           | 0.9800       |                   |              |
| O11—Er1—O11C       | 72.5 (3)     | N3C—C3C—C2C        | 117.7 (9)    |                   |              |
| O11—Er1—O12A       | 74.7 (2)     | N3C—C3C—C4C        | 120.7 (10)   |                   |              |
| O11—Er1—O12B       | 80.6 (2)     | C2C—C3C—C4C        | 121.5 (9)    |                   |              |
| O11—Er1—O12C       | 77.0 (3)     | C14A—C4A—C3A       | 124.2 (9)    |                   |              |
| O11—Er1—O11A       | 140.9 (3)    | C14A—C4A—C5A       | 117.3 (9)    |                   |              |
| O11—Er1—O11B       | 143.3 (2)    | C3A—C4A—C5A        | 118.5 (11)   |                   |              |
| O11C—Er1—O12A      | 145.4 (2)    | C14B—C4B—C3B       | 124.4 (8)    |                   |              |
| O11C—Er1—O12B      | 84.1 (2)     | C14B—C4B—C5B       | 116.3 (8)    |                   |              |
| O11C—Er1—O12C      | 94.7 (2)     | C3B—C4B—C5B        | 119.3 (10)   |                   |              |
| O11A—Er1—O11C      | 73.9 (2)     | C14C—C4C—C3C       | 121.1 (8)    |                   |              |
| O11B—Er1—O11C      | 130.3 (2)    | C14C—C4C—C5C       | 119.7 (9)    |                   |              |
| O12A—Er1—O12B      | 80.0 (2)     | C3C—C4C—C5C        | 119.2 (10)   |                   |              |
| O12A—Er1—O12C      | 88.4 (2)     | C4A—C5A—C6A        | 121.0 (10)   |                   |              |
| O11A—Er1—O12A      | 130.5 (2)    | C4B—C5B—C6B        | 120.5 (10)   |                   |              |
| O11B—Er1—O12A      | 83.5 (2)     | C4C—C5C—C6C        | 120.1 (10)   |                   |              |
| O12B—Er1—O12C      | 156.8 (2)    | C1A—C6A—C5A        | 119.9 (9)    |                   |              |
| O11A—Er1—O12B      | 76.7 (2)     | C1B—C6B—C5B        | 119.3 (9)    |                   |              |
| O11B—Er1—O12B      | 124.6 (2)    | C1C—C6C—C5C        | 120.3 (9)    |                   |              |
| O11A—Er1—O12C      | 125.3 (2)    | O11A—C11A—O12A     | 127.7 (8)    |                   |              |
| O11B—Er1—O12C      | 73.2 (2)     | O11A—C11A—C1A      | 116.0 (7)    |                   |              |
| O11A—Er1—O11B      | 75.2 (2)     | O12A—C11A—C1A      | 116.3 (8)    |                   |              |
| O11—S1—C11         | 103.9 (5)    | O11B—C11B—O12B     | 121.6 (8)    |                   |              |
| O11—S1—C12         | 106.0 (5)    | O11B—C11B—C1B      | 119.8 (8)    |                   |              |
| C11—S1—C12         | 99.3 (5)     | O12B—C11B—C1B      | 118.6 (8)    |                   |              |
| Er1—O11—S1         | 133.1 (4)    | O11C—C11C—O12C     | 123.6 (9)    |                   |              |
| Er1—O11A—C11A      | 140.3 (6)    | O11C—C11C—C1C      | 118.1 (8)    |                   |              |
| Er1—O11B—C11B      | 110.9 (5)    | O12C—C11C—C1C      | 118.3 (8)    |                   |              |
Er1—O11C—C11C 113.9 (6) C1A—C2A—H2A 120.00
Er1—O12A—C11A 132.8 (6) C3A—C2A—H2A 120.00
Er1—O12B—C11B 172.3 (6) C1B—C2B—H2B 120.00
Er1'—O12C—C11C 128.2 (6) C3B—C2B—H2B 120.00
O31A—N3A—O32A 124.3 (12) C1C—C2C—H2C 120.00
O31A—N3A—C3A 118.5 (10) C3C—C2C—H2C 120.00
O32A—N3A—C3A 117.1 (12) C4A—C5A—H5A 119.00
O31B—N3B—O32B 118.3 (13) C6A—C5A—H5A 120.00
O31B—N3B—C3B 120.3 (12) C4B—C5B—H5B 120.00
O32B—N3B—C3B 121.2 (11) C6B—C5B—H5B 120.00
O31C—N3C—O32C 124.1 (12) C4C—C5C—H5C 120.00
O31C—N3C—C3C 116.7 (11) C6C—C5C—H5C 120.00
O32C—N3C—C3C 119.2 (10) C1A—C6A—H6A 120.00
C2A—C1A—C6A 119.6 (9) C5A—C6A—H6A 120.00
C2A—C1A—C11A 120.3 (8) C1B—C6B—H6B 120.00
C6A—C1A—C11A 120.1 (8) C5B—C6B—H6B 120.00
C2B—C1B—C6B 119.5 (9) C1C—C6C—H6C 120.00
C2B—C1B—C11B 121.6 (8) C5C—C6C—H6C 120.00
C6B—C1B—C11B 118.9 (8) S1—C11—H111 109.00
C2C—C1C—C6C 119.0 (9) S1—C11—H112 109.00
C2C—C1C—C11C 120.7 (8) S1—C11—H113 109.00
C6C—C1C—C11C 120.3 (8) H111—C11—H112 109.00
H111—C11—H113 110.00
C1A—C2A—C3A 119.6 (9) H112—C11—H113 110.00
C1B—C2B—C3B 120.6 (9) H112—C11—H113 110.00
C1C—C2C—C3C 119.8 (9) S1—C12—H121 109.00
N3A—C3A—C2A 115.0 (10) S1—C12—H122 109.00
N3A—C3A—C4A 123.6 (10) S1—C12—H123 109.00
C2A—C3A—C4A 121.4 (10) H121—C12—H122 110.00
N3B—C3B—C2B 116.5 (9) H121—C12—H123 110.00
N3B—C3B—C4B 122.7 (10) H122—C12—H123 109.00
C2B—C3B—C4B 120.7 (9)

O11C—Er1—O11—S1 123.8 (6) O31C—N3C—C3C—C2C −58.0 (16)
O12A—Er1—O11—S1 −67.4 (6) C6A—C1A—C11A—O11A −20.4 (13)
O12B—Er1—O11—S1 −149.5 (6) C2A—C1A—C11A—O11A 158.7 (9)
O12C—Er1—O11—S1 24.6 (5) C2A—C1A—C11A—O12A −20.0 (13)
O11A'—Er1—O11—S1 155.6 (4) C2A—C1A—C6A—C5A −0.8 (16)
O11B'—Er1—O11—S1 −11.7 (8) C11A—C1A—C6A—C5A 178.3 (10)
O11—Er1—O11C—C11C −136.0 (7) C11A—C1A—C2A—C3A −175.0 (9)
O12A—Er1—O11C—C11C −155.2 (6) C6A—C1A—C2A—C3A 4.1 (15)
O12B—Er1—O11C—C11C 142.1 (6) C6A—C1A—C11A—O12A 160.9 (9)
O12C—Er1—O11C—C11C −61.2 (6) C2B—C1B—C11B—O12B −4.0 (14)
O11A''—Er1—O11C—C11C 64.2 (6) C2B—C1B—C11B—O11B 177.2 (9)
O11B''—Er1—O11C—C11C 10.6 (7) C6B—C1B—C11B—O11B −4.2 (14)
O11—Er1—O12A—C11A −102.6 (8) C11B—C1B—C6B—C5B −177.4 (9)
O11C—Er1—O12A—C11A −83.6 (8) C2B—C1B—C6B—C5B 1.2 (15)
O12B—Er1—O12A—C11A −19.8 (7) C6B—C1B—C2B—C3B 0.5 (16)
O12C—Er1—O12A—C11A −179.6 (8) C11B—C1B—C2B—C3B 179.1 (10)
O11A\textsuperscript{ii}—Er1—O12A—C11A 43.0 (8) C6B—C1B—C11B—O12B 174.7 (9)
O11B—Er1—O12A—C11A 107.2 (8) C6C—C1C—C11C—O11C −18.6 (13)
O11—Er1—O12C—C11C\textsuperscript{i} −162.8 (8) C2C—C1C—C11C—O11C 160.3 (8)
O11C—Er1—O12C—C11C\textsuperscript{i} 126.4 (8) C6C—C1C—C11C—O12C −179.3 (9)
O11− Er1—O11A\textsuperscript{ii}—C11A\textsuperscript{ii} 85.7 (10) C2C—C1C—C6C—C5C 1.5 (14)
O11C—Er1—O11A\textsuperscript{ii}—C11A\textsuperscript{ii} 117.2 (10) C6C—C1C—C2C—C3C −0.5 (14)
O11− Er1—O11B\textsuperscript{ii}—C11B\textsuperscript{ii} −118.1 (6) C1A—C2A—C3A—N3A 173.0 (10)
O11C—Er1—O11B\textsuperscript{ii}—C11B\textsuperscript{ii} 123.2 (6) C1B—C2B—C3B—C4B −3.0 (17)
O12A—Er1—O11A\textsuperscript{ii}—C11A\textsuperscript{ii} −34.5 (11) C11C—C1C—C6C—C5C −179.6 (9)
O12B—Er1—O11A\textsuperscript{ii}—C11A\textsuperscript{ii} 29.6 (9) C1A—C2A—C3A—N3A 173.0 (10)

Symmetry codes: (i) −x, −y+1, −z+1; (ii) −x+1, −y+1, −z+1.

**Hydrogen-bond geometry (Å, °)**

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| C2A—H2A···S1 | 0.95 | 2.86 | 3.743 (10) | 155 |
| C2B—H2B···O11 | 0.95 | 2.56 | 3.298 (13) | 135 |
| C11—H111···C14A\textsuperscript{ii} | 0.98 | 2.79 | 3.486 (11) | 129 |
| C12—H123···O32A\textsuperscript{iv} | 0.98 | 2.44 | 3.376 (15) | 158 |

Symmetry codes: (iii) −x+1, −y+1, −z; (iv) −x, −y+1, −z.

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