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Crystal structure of (diaqua-bis(phenanthroline-κ^2N,N')-tetrakis(μ_2-3,4,5,6-tetrafluorophthalato-$\kappa^4O,O:O':O''$;$\kappa^2O:O'$)dierbium (III) phenanthroline (1/2), $C_{80}H_{38}Er_2F_{16}N_8O_{18}$

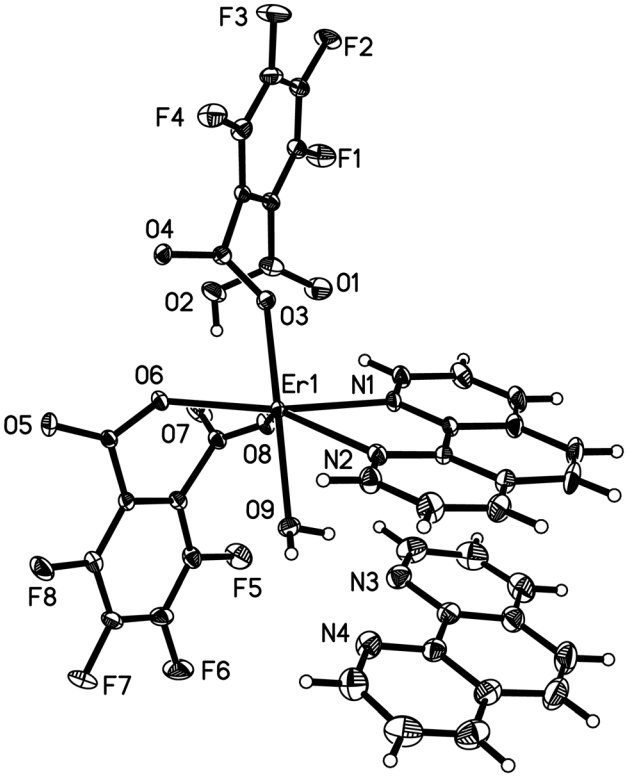

Figure: The molecular structure of the title compound, showing the atom numbering scheme. Displacement ellipsoids are shown at the 30% probability level.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.20 × 0.19 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	2.49 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	29.1°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	15,821, 8127, 0.034
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 7495
$N(param)_{refined}$:	561
Programs:	CrysAlis^{Pro} [1], Olex2 [2], Shelx [3, 4]

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Abstract

$C_{80}H_{38}Er_2F_{16}N_8O_{18}$, triclinic, $P\bar{1}$ (no. 2), a = 11.4228(4) Å, b = 12.6712(3) Å, c = 13.2663(4) Å, α = 102.660(2)°, β = 98.047(3)°, γ = 105.509(3)°, V = 1764.54(10) Å^3, Z = 1, $R_{gt}(F)$ = 0.0311, $wR_{ref}(F^2)$ = 0.0668, T = 293(2) K.

CCDC no.: 2235105

The asymmetric unit of the title crystal structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

1 Source of materials

A mixture of $Er(NO_3)_3{\cdot}6H_2O$ (0.0923 g, 0.2 mmol), 3,4,5,6-tetrafluorophthalic acid (0.054 g, 0.2 mmol) and phenanthroline (35.7 mg, 0.15 mmol) were dissolved in 8 mL of deionized water. The mixture was sealed in a 25 mL Teflon-lined steel autoclave after ultrasound treatment for 15 min and heated at 120 °C for 72 h. The mixture was cooled to room temperature at a rate of 2 °C/h, and colorless block crystals were isolated by filtration, washed with distilled water and dried in air (CCDC number 2235105).

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Er1	0.58494 (2)	0.16248 (2)	0.55111 (2)	0.01820 (5)
F1	0.5363 (2)	0.19806 (18)	0.00720 (16)	0.0467 (6)
F2	0.2935 (2)	0.12846 (17)	−0.08053 (16)	0.0440 (5)
F3	0.1286 (2)	−0.0091 (2)	0.00034 (18)	0.0541 (6)
F4	0.20538 (19)	−0.07264 (16)	0.17039 (16)	0.0411 (5)
F5	1.0644 (2)	0.24678 (17)	0.56698 (19)	0.0500 (6)
F6	1.1730 (2)	0.18881 (19)	0.73143 (19)	0.0528 (6)
F7	1.0458 (2)	0.00672 (18)	0.79144 (16)	0.0429 (5)
F8	0.8218 (2)	−0.12321 (16)	0.68203 (17)	0.0419 (5)
O1	0.6936 (2)	0.2511 (2)	0.2149 (2)	0.0474 (7)
O2	0.6749 (2)	0.0697 (2)	0.2140 (2)	0.0397 (6)
H2	0.7401	0.0965	0.2585	0.060*
O3	0.5078 (2)	0.11221 (16)	0.36582 (16)	0.0247 (5)
O4	0.4227 (2)	−0.07749 (16)	0.30500 (17)	0.0278 (5)
O5	0.6461 (2)	−0.17597 (16)	0.49578 (19)	0.0323 (6)
O6	0.61447 (19)	−0.01466 (16)	0.48870 (17)	0.0217 (5)
O7	0.8569 (2)	0.1082 (2)	0.36340 (19)	0.0392 (6)
O8	0.7554 (2)	0.19275 (16)	0.46982 (17)	0.0251 (5)
O9	0.7677 (2)	0.24310 (18)	0.68284 (18)	0.0297 (5)
H9A	0.7512	0.2435	0.7452	0.044*
H9B	0.7995	0.3151	0.6872	0.044*
N1	0.6284 (2)	0.35821 (19)	0.5296 (2)	0.0231 (6)
N2	0.5334 (3)	0.3014 (2)	0.6946 (2)	0.0253 (6)
C1	0.4992 (3)	0.1074 (2)	0.1432 (2)	0.0235 (7)
C2	0.4139 (3)	0.0375 (2)	0.1865 (2)	0.0221 (7)
C3	0.2922 (3)	−0.0037 (3)	0.1341 (3)	0.0291 (7)
C4	0.2498 (3)	0.0265 (3)	0.0447 (3)	0.0325 (8)
C5	0.3326 (3)	0.0963 (3)	0.0043 (3)	0.0301 (8)
C6	0.4564 (3)	0.1335 (3)	0.0517 (3)	0.0269 (7)
C7	0.6341 (3)	0.1526 (3)	0.1950 (3)	0.0306 (8)
C8	0.4535 (3)	0.0226 (2)	0.2951 (2)	0.0215 (6)
C9	0.8190 (3)	−0.0048 (2)	0.5654 (2)	0.0210 (6)
C10	0.8824 (3)	0.0925 (2)	0.5377 (2)	0.0228 (7)
C11	1.0020 (3)	0.1529 (3)	0.5930 (3)	0.0288 (7)
C12	1.0584 (3)	0.1255 (3)	0.6776 (3)	0.0325 (8)
C13	0.9959 (3)	0.0321 (3)	0.7062 (3)	0.0287 (7)
C14	0.8785 (3)	−0.0331 (3)	0.6489 (3)	0.0263 (7)
C15	0.6865 (3)	−0.0721 (2)	0.5130 (2)	0.0199 (6)
C16	0.8259 (3)	0.1341 (2)	0.4496 (3)	0.0235 (7)
C17	0.4821 (3)	0.2743 (3)	0.7726 (3)	0.0332 (8)
H17	0.4619	0.1990	0.7754	0.040*
C18	0.4567 (4)	0.3530 (3)	0.8508 (3)	0.0421 (10)
H18	0.4203	0.3300	0.9040	0.051*
C19	0.4853 (4)	0.4634 (3)	0.8487 (3)	0.0414 (9)
H19	0.4684	0.5165	0.9004	0.050*
C20	0.5697 (4)	0.6106 (3)	0.7587 (3)	0.0446 (10)
H20	0.5548	0.6663	0.8092	0.054*
C21	0.6181 (4)	0.6391 (3)	0.6791 (3)	0.0404 (9)
H21	0.6374	0.7141	0.6756	0.049*
C22	0.6894 (3)	0.5814 (3)	0.5127 (3)	0.0372 (9)
H22	0.7103	0.6556	0.5068	0.045*
C23	0.7055 (3)	0.4974 (3)	0.4383 (3)	0.0368 (9)
H23	0.7375	0.5134	0.3807	0.044*
C24	0.6737 (3)	0.3865 (3)	0.4489 (3)	0.0297 (8)
H24	0.6847	0.3297	0.3969	0.036*
C25	0.6124 (3)	0.4420 (2)	0.6046 (3)	0.0227 (7)
C26	0.6408 (3)	0.5552 (2)	0.5987 (3)	0.0306 (8)
C27	0.5400 (3)	0.4964 (3)	0.7688 (3)	0.0318 (8)
C28	0.5616 (3)	0.4121 (2)	0.6914 (2)	0.0227 (7)
N3	0.9074 (3)	0.4650 (2)	0.7305 (2)	0.0395 (7)
N4	0.8259 (3)	0.3905 (2)	0.8968 (3)	0.0411 (8)
C29	0.7941 (4)	0.3579 (4)	0.9799 (3)	0.0509 (11)
H29	0.7723	0.2807	0.9755	0.061*
C30	0.7914 (4)	0.4322 (4)	1.0737 (4)	0.0599 (12)
H30	0.7689	0.4050	1.1302	0.072*
C31	0.8224 (4)	0.5450 (4)	1.0805 (4)	0.0581 (12)
H31	0.8207	0.5962	1.1419	0.070*
C32	0.8908 (4)	0.7022 (4)	0.9971 (4)	0.0609 (13)
H32	0.8864	0.7551	1.0561	0.073*
C33	0.9282 (4)	0.7372 (3)	0.9159 (4)	0.0584 (12)
H33	0.9484	0.8142	0.9191	0.070*
C34	0.9848 (4)	0.6949 (4)	0.7406 (4)	0.0543 (12)
H34	1.0086	0.7716	0.7425	0.065*
C35	0.9951 (4)	0.6181 (4)	0.6583 (4)	0.0592 (12)
H35	1.0295	0.6412	0.6042	0.071*
C36	0.9537 (4)	0.5027 (4)	0.6543 (3)	0.0523 (11)
H36	0.9588	0.4500	0.5954	0.063*
C37	0.9010 (3)	0.5426 (3)	0.8171 (3)	0.0323 (8)
C38	0.9380 (4)	0.6597 (3)	0.8245 (3)	0.0425 (9)
C39	0.8572 (4)	0.5842 (3)	0.9947 (3)	0.0443 (10)
C40	0.8593 (3)	0.5036 (3)	0.9042 (3)	0.0342 (8)

2 Experimental details

Using Olex2 [2], the structure was solved with the ShelXT [3] structure solution program and refined with the ShelXL [4] refinement package. The carbon bound hydrogen atoms were placed in calculated positions and refined using a riding model on attached atoms.

3 Comment

In recent decades, a large number of Er(III) complexes have attracted considerable attentions due to their structural diversity and potential applications in many areas [5–8]. Er(III)–MOFs can be constructed from Er(III) ions and carboxylate and nitrogen-containing ligands. Among rod N-donor ligands, 1,10-phenanthroline (1,10–Phen) has been extensively used in the preparation of Er complexes [5–7]. However, tetrafluorophthalic acid (H_2TFPA), as a common derivative of phthalic acid, is rarely used in the assembly process [8]. In this work, a binuclear Er(III) complex was

constructed with TFPA and 1,10–Phen as mixed ligands. The asymmetric unit contains one Er(III), two tetrafluorophthalate ($HTFPA^-$), one coordinated water molecule, one coordinated 1,10–Phen, and one free 1,10–Phen molecule (see Figure). Er1 is nine-coordinated by two N atoms (N1, N2) of phen ligand, one O atom (O9) from coordinated water molecule, two O atoms (O3, O4#1, #1: $1 - x, -y, 1 - z$) from two $HTFPA^-$ ligands and four O atoms (O6, O8, O5#1 and O6#1) from two $TFPA^{2-}$ ligands. The Er–N bond lengths are 2.484(2) and 2.543(2) Å and the Er–O bond lengths are in the range of 2.339(2)–2.686(2) Å, respectively. Two $HTFPA^-$ and two $TFPA^{2-}$ ligands adopt bis-monodentate and bis-bidentate bridging modes, respectively, to link neighboring Er(III) ions into a nuclear $\{Er_2\}$ unit.

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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