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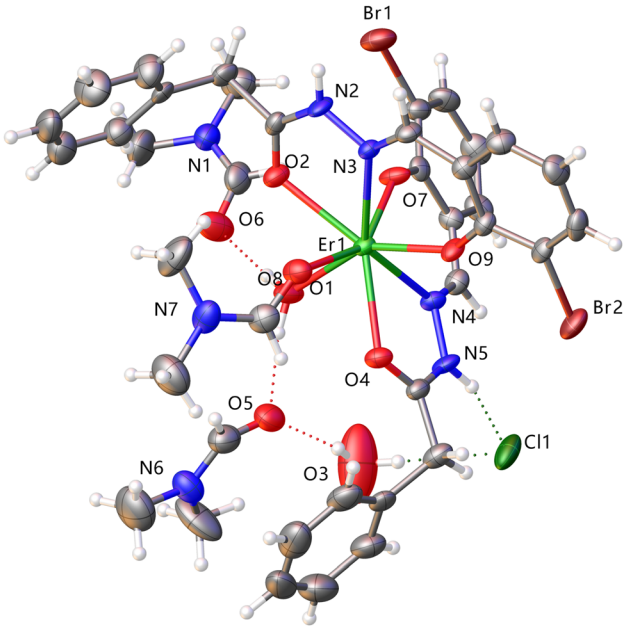
The crystal structure of aqua-bis{2-bromo-6-((2-(2-phenylacetyl)hydrazineylidene)methyl) phenolato-κ^3*N*,*O*,*O*′}-dimethylformamide-κ^1*O*-erbium(III) chloride – dimethylformamide – water (1/2/1), $C_{39}H_{49}N_7O_9Br_2ClEr$



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Abstract

$C_{39}H_{49}N_7O_9Br_2ClEr$, triclinic, $P\bar{1}$ (no. 2), a = 12.3164(9) Å, b = 12.7581(9) Å, c = 14.5023(11) Å, α = 70.873(2)°, β = 80.281(2)°, γ = 65.810(2)°, V = 1962.4(3) Å^3, Z = 1, $R_{gt}(F)$ = 0.0383, $wR_{ref}(F^2)$ = 0.0765, T = 296.15 K.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.14 × 0.11 × 0.09 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	2.46 mm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD 6000, ω
θ_{max}, completeness:	25.1°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	44,757, 6893, 0.064
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 5870
$N(param)_{refined}$:	536
Programs:	Bruker [1], Olex2 [2], SHELX [3], Diamond [4]

The molecular 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

The title Er(III) complex was synthesized using the following one-pot method: 0.1502 g phenacetic acid hydrazide (0.1 mmol), 0.2010 g 3-bromo-2-hydroxybenzaldehyde (1.0 mmol), 0.1909 g $ErCl_3{\cdot}6H_2O$ (0.50 mmol), and 0.040 g NaOH (1.0 mmol) were added to the 25 mL ethanol–water–dimethylformamide (v:v:v = 3:1:1) solution with stirring. The mixture was continued to react for 6 h at 80 °C and then cooled to room temperature. After filtering the mixture, the filtrate was allowed to slowly volatilization at room temperature. The yellow block crystals of the title compound were received in 30 days. **Anal. Calcd.** for $C_{39}H_{49}N_7O_9Br_2ClEr$: C, 41.70; H, 4.37; N, 8.73. Found: C, 41.89; H, 4.01; N, 8.52.

2 Experimental details

The hydrogen atoms were positioned geometrically (C–H = 0.93–0.96 Å, N–H = 0.86 Å and O–H = 0.85 Å). Their U_{iso} values were set to 1.2U_{eq} or 1.5U_{eq} of the parent atoms.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.8902 (4)	0.1835 (4)	0.4217 (3)	0.0281 (11)
C01B	0.7476 (6)	1.1388 (6)	−0.1487 (4)	0.0561 (17)
C2	0.9117 (5)	0.0809 (4)	0.3945 (4)	0.0346 (13)
C3	1.0195 (5)	0.0324 (5)	0.3457 (4)	0.0445 (15)
H3	1.036039	−0.035343	0.326178	0.053*
C4	1.0997 (5)	0.0847 (5)	0.3270 (4)	0.0433 (14)
H4	1.172532	0.052292	0.295603	0.052*
C5	1.0726 (5)	0.1873 (5)	0.3550 (4)	0.0364 (13)
H5	1.128520	0.222604	0.340741	0.044*
C6	0.8223 (6)	0.0295 (5)	0.4192 (5)	0.0517 (16)
H6	0.835014	−0.037817	0.400443	0.062*
C7	0.7223 (6)	0.0763 (5)	0.4680 (5)	0.0535 (17)
H7	0.666556	0.040719	0.483303	0.064*
C8	0.6984 (5)	0.1794 (5)	0.4972 (4)	0.0379 (13)
C9	0.7815 (4)	0.2352 (4)	0.4731 (3)	0.0283 (11)
C10	0.6614 (4)	0.3838 (5)	0.5432 (3)	0.0311 (12)
H10	0.647295	0.453679	0.558474	0.037*
C11	0.5743 (5)	0.3346 (5)	0.5706 (4)	0.0403 (14)
H11	0.504683	0.370385	0.604166	0.048*
C12	0.5932 (5)	0.2337 (6)	0.5473 (4)	0.0487 (16)
H12	0.535691	0.200215	0.564789	0.058*
C13	0.7568 (5)	0.5764 (5)	0.3036 (4)	0.0303 (12)
C14	0.6755 (4)	0.6567 (5)	0.2192 (4)	0.0366 (13)
H14A	0.605586	0.637212	0.227269	0.044*
H14B	0.649683	0.740038	0.217678	0.044*
C15	0.6849 (5)	0.7079 (5)	0.0451 (4)	0.0350 (13)
C16	0.5700 (5)	0.7894 (5)	0.0354 (4)	0.0495 (16)
H16	0.520653	0.801581	0.090173	0.059*
C17	0.5287 (5)	0.8535 (5)	−0.0579 (4)	0.0540 (16)
H17	0.450330	0.908324	−0.064871	0.065*
C18	0.5997 (5)	0.8387 (5)	−0.1405 (4)	0.0409 (14)
C19	0.7166 (5)	0.7561 (5)	−0.1281 (4)	0.0378 (13)
H19	0.766553	0.745098	−0.182741	0.045*
C20	0.7591 (5)	0.6912 (5)	−0.0376 (4)	0.0370 (13)
H20	0.837352	0.635982	−0.030637	0.044*
C21	0.5579 (6)	0.9029 (6)	−0.2414 (5)	0.0605 (18)
C22	0.4297 (8)	0.9878 (9)	−0.2550 (6)	0.131 (4)
H22A	0.415346	1.023191	−0.323458	0.196*
H22B	0.413340	1.049826	−0.224921	0.196*
H22C	0.378685	0.944609	−0.225183	0.196*
C23	0.9526 (4)	0.7049 (4)	0.3744 (3)	0.0263 (11)
C24	0.9001 (5)	0.7596 (4)	0.2755 (4)	0.0391 (14)
H24A	0.938076	0.704223	0.236469	0.047*
H24B	0.815694	0.774631	0.282447	0.047*
C25	0.8752 (5)	0.9278 (5)	0.1356 (4)	0.0413 (14)
C26	0.8770 (6)	1.0419 (5)	0.0976 (4)	0.0589 (18)
H26	0.905826	1.072260	0.133922	0.071*
C27	0.8361 (6)	1.1098 (5)	0.0060 (4)	0.0595 (18)
H27	0.836077	1.186939	−0.018401	0.071*
C28	0.7950 (5)	1.0669 (5)	−0.0510 (4)	0.0410 (14)
C29	0.7964 (5)	0.9515 (5)	−0.0123 (4)	0.0458 (15)
H29	0.769054	0.920363	−0.049083	0.055*
C30	0.8373 (5)	0.8824 (5)	0.0791 (4)	0.0473 (15)
H30	0.839362	0.804463	0.102887	0.057*
C31	0.7543 (8)	1.2601 (6)	−0.1950 (5)	0.086 (2)
H31A	0.722513	1.293400	−0.258935	0.128*
H31B	0.835911	1.252224	−0.200264	0.128*
H31C	0.708895	1.312266	−0.155437	0.128*
C32	0.8198 (4)	0.5562 (4)	0.6351 (3)	0.0233 (11)
C33	0.7224 (4)	0.5834 (5)	0.7126 (4)	0.0341 (12)
H33A	0.748977	0.526125	0.775489	0.041*
H33B	0.703777	0.663110	0.717281	0.041*
C34	0.5227 (4)	0.5928 (4)	0.7509 (3)	0.0298 (12)
C35	0.5207 (4)	0.5993 (5)	0.8441 (4)	0.0383 (13)
H35	0.588194	0.595306	0.867948	0.046*
C36	0.4175 (5)	0.6118 (5)	0.9018 (4)	0.0389 (13)
H36	0.416530	0.615863	0.964784	0.047*
C37	0.3150 (4)	0.6186 (4)	0.8682 (4)	0.0318 (12)
C38	0.3184 (4)	0.6158 (5)	0.7737 (4)	0.0359 (13)
H38	0.249938	0.623752	0.748488	0.043*
C39	0.4207 (4)	0.6014 (5)	0.7161 (4)	0.0372 (13)
H39	0.421547	0.597513	0.653031	0.045*
C40	0.2085 (5)	0.6253 (5)	0.9346 (5)	0.0399 (14)
C41	0.0923 (5)	0.6615 (6)	0.8918 (5)	0.0608 (18)
H41A	0.094526	0.599346	0.867162	0.091*
H41B	0.077769	0.734455	0.839387	0.091*
H41C	0.029683	0.674089	0.941207	0.091*
N1	0.7624 (3)	0.3364 (3)	0.4968 (3)	0.0241 (9)
N2	0.9713 (3)	0.2373 (3)	0.4007 (3)	0.0276 (9)
O1	0.8125 (3)	0.5002 (3)	0.5821 (2)	0.0260 (7)
O2	0.9015 (3)	0.5922 (3)	0.6327 (2)	0.0342 (8)
O3	0.6192 (3)	0.5764 (3)	0.6877 (2)	0.0382 (9)
O4	0.2159 (3)	0.6014 (4)	1.0220 (3)	0.0573 (11)
O5	0.9376 (3)	0.6092 (3)	0.4262 (2)	0.0247 (7)
O6	1.0093 (3)	0.7475 (3)	0.4040 (2)	0.0328 (8)
O7	0.9162 (4)	0.8685 (3)	0.2278 (3)	0.0499 (11)
O8	0.7010 (5)	1.1016 (4)	−0.1916 (3)	0.0879 (17)
O9	0.6224 (4)	0.8850 (5)	−0.3117 (3)	0.0786 (15)
O10	0.7390 (3)	0.6389 (3)	0.1311 (3)	0.0456 (10)
O11	0.8579 (3)	0.5004 (3)	0.2889 (2)	0.0345 (8)
O12	0.7201 (3)	0.5891 (3)	0.3862 (2)	0.0363 (9)
O14	1.0169 (8)	0.5585 (11)	0.1300 (8)	0.220 (5)
H14C	1.087155	0.514904	0.115454	0.331*
H14D	0.997885	0.514963	0.183153	0.331*
Yb1	0.91664 (2)	0.42751 (2)	0.45313 (2)	0.02433 (8)
O13	0.6677 (8)	0.8076 (8)	0.4780 (6)	0.139 (2)*
H13A	0.736095	0.809539	0.476669	0.208*
H13B	0.622840	0.839166	0.521038	0.208*

3 Comment

Erbium complexes may show novel structures [5] and excellent properties in catalysis [6], dual visible/near-infrared luminescence [7], luminescent electrochromic device [8], single-molecule magnet [9]. As an important class of polypodand, aroylhydrazone ligands have been used to construct diverse metal complexes [10]. We have been working on the synthesis, structure and properties of aroylhydrazone metal

complexes [11–16]. In this work, we report a new aroylhydrazone Er(III) complex based on phenacetic acid hydrazide, 3-bromo-2-hydroxybenzaldehyde, $ErCl_3{\cdot}6H_2O$, and NaOH as starting materials.

The molecular structure of Er(III) complex is shown in the figure. The asymmetric unit of the title compound consists of one Er(III) ion, two 2 monoanionic hydrazone ligands, one coordinated dimethylformamide molecule, one coordinated water molecule, two uncoordinated dimethylformamide molecules, one uncoordinated water molecule, and one chloride ion. The Er(III) atom is eight-coordinated by four oxygen atoms (O2, O4, O7, O9) and two nitrogen atoms (N3, N4) from two different tridentate 2-bromo-6-(phenylacetyl-hydrazonomethyl)-phenylate ligands, one oxygen atom (O8) from one coordinated dimethylformamide molecule, one oxygen atom (O1) from one coordinated water molecule. The bond lengths of Er–O and Er–N are in the range of 2.184(4)–2.388(4) Å and 2.491(4)–2.506(5) Å, respectively. The dihedral angles of plane 1 (C2–C3–C4–C5–C6–C14) and plane 2 (C1–C13–C12–C11–C10–C15), plane 3 (C16–C17–C18–C19–C20–C21) and plane 4 (C25–C26–C27–C28–C29–C30) are 61.41° and 56.67°, respectively.

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