Crystal structure of bis[(1,10-phenanthroline-*N*,*N*')(2-bromobenzoato)-bis(μ-2-bromobenzoato)holmium(III)], $[Ho(C_{12}H_8N_2)(BrC_7H_4O_2)_3]_2$

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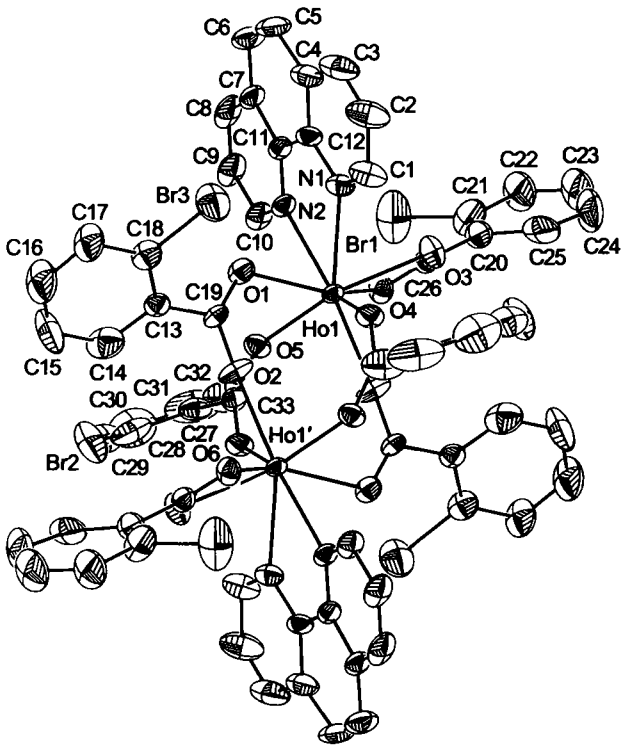

Abstract

$C_{66}H_{40}Br_6Ho_2N_4O_{12}$, triclinic, $P\bar{1}$ (no. 2), a = 11.368(2) Å, b = 12.212(2) Å, c = 13.944(3) Å, α = 110.73(3)°, β = 105.02(3)°, γ = 104.02(3)°, V = 1624.8 Å^3, Z = 1, $R_{gt}(F)$ = 0.056, $wR_{ref}(F^2)$ = 0.171, T = 293 K.

Source of material

Ho_2O_3 (0.09 g, 0.24 mmol), 1,10-phenanthroline (phen · H_2O; 0.05 g, 0.25 mmol), 2-bromo-benzoic acid (0.10 g, 0.50 mmol), 15 mL CH_3OH/H_2O (1:2 *v*/*v*) were mixed and stirred for ca. 1 h. Subsequently, the resulting suspension was heated in a 23 ml Teflon-lined stainless-steel autoclave at 423 K for 7 days. After the autoclave was cooled to room temperature, yellowish pillar-like crystals were obtained.

Discussion

The crystal structure of title compound is related to the structure of $[Gd_2(C_{12}H_8N_2)_2(C_6H_5COO)_6]$ [1] and similar to the structure of $[Pb(C_7H_4O_2I)(C_{12}H_8N_2)_2](C_7H_4O_2I) \cdot 2H_2O$ [2].

Within the $[Ho(BrC_6H_4COO)(phen)(\mu\text{-}BrC_6H_4COO)_{4/2}]_2$ complex molecules, the Ho atoms are each coordinated by two N atoms from one bidentately chelating *o*-phenanthroline ligand and six O atoms from five 2-bromo-benzoic acid anions ligands, to complete a significantly distorted PbN_2O_6 polyhedron environment with d(Ho—N) = 2.515(6) Å and 2.555(7) Å, d(Ho—O) = 2.249(6) Å – 2.437(5) Å. Four carboxy groups of 2-bromobenzoic acid anions bridge Ho and Ho^i atoms (i: $1-x,-y,1-z$) to a dinuclear complex. Moreover, the dinuclear molecules, $[Ho(BrC_6H_4COO)(phen)(BrC_6H_4COO)_{4/2})]_2$, are connected to each other via weak hydrogen bonds between the 2-bromobenzoic acid anion O atoms and *o*-phenanthroline C atoms with d(C8–H8···O2^{ii}) = 3.35(5) Å, ∠C8–H8···O2^{ii} = 150.3(8)° (ii: $1-x,1-y,1-z$). Through the weak hydrogen interactions the compound is interlinked to forms the 1D supramolecular chains along the [010] direction.

Table 1. Data collection and handling.

Crystal:	yellowish, pillar-like, size 0.10 × 0.20 × 0.34 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	61.70 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX CCD II, φ/ω
$2\theta_{max}$:	57.38°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	25743, 8231
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 6589
$N(param)_{refined}$:	407
Programs:	SHELXS-97 [3], SHELXL-97 [4]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	0.5810	0.0348	0.2071	0.111
H(2)	2*i*	0.6711	0.1372	0.1165	0.141
H(3)	2*i*	0.6456	0.3156	0.1252	0.130
H(5)	2*i*	0.5686	0.5019	0.2114	0.125
H(6)	2*i*	0.4505	0.5730	0.3061	0.115
H(8)	2*i*	0.3093	0.5421	0.4102	0.124
H(9)	2*i*	0.2191	0.4303	0.4915	0.103
H(10)	2*i*	0.2655	0.2538	0.4823	0.081
H(14)	2*i*	0.7950	0.3166	0.7901	0.130
H(15)	2*i*	0.9810	0.4845	0.9135	0.171
H(16)	2*i*	1.1244	0.5825	0.8530	0.165
H(17)	2*i*	1.0787	0.5180	0.6673	0.128
H(22)	2*i*	–0.2357	–0.0441	0.0397	0.164
H(23)	2*i*	–0.2622	–0.2140	–0.1191	0.169
H(24)	2*i*	–0.1161	–0.3137	–0.1293	0.179
H(25)	2*i*	0.0954	–0.2225	0.0131	0.197
H(29)	2*i*	0.3930	0.3747	0.9667	0.147
H(30)	2*i*	0.1772	0.3351	0.8874	0.186
H(31)	2*i*	0.0566	0.2095	0.7000	0.193
H(32)	2*i*	0.1639	0.1140	0.5896	0.135

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ho(1)	2*i*	0.41498(3)	0.05978(3)	0.38521(2)	0.0462(2)	0.0320(2)	0.0465(2)	0.0177(1)	0.0116(1)	0.0214(1)
O(1)	2*i*	0.3952(5)	0.1397(5)	0.5551(4)	0.068(3)	0.069(4)	0.055(3)	0.037(3)	0.022(3)	0.035(3)
O(2)	2*i*	0.6233(5)	0.2228(5)	0.4995(5)	0.048(3)	0.054(3)	0.076(4)	0.016(2)	0.012(3)	0.030(3)
O(3)	2*i*	0.2641(6)	−0.0522(7)	0.1992(5)	0.065(4)	0.093(5)	0.054(3)	0.027(3)	0.011(3)	0.021(3)
O(4)	2*i*	0.1792(5)	0.0106(6)	0.3215(5)	0.050(3)	0.064(4)	0.063(3)	0.019(3)	0.008(2)	0.022(3)
O(5)	2*i*	0.3408(7)	−0.1138(6)	0.4081(7)	0.090(4)	0.051(3)	0.131(6)	0.032(3)	0.057(4)	0.059(4)
O(6)	2*i*	0.5448(6)	−0.0510(5)	0.3348(5)	0.081(4)	0.062(3)	0.065(3)	0.048(3)	0.030(3)	0.037(3)
N(1)	2*i*	0.5072(7)	0.1569(7)	0.2728(6)	0.064(4)	0.072(5)	0.067(4)	0.031(4)	0.023(3)	0.047(4)
N(2)	2*i*	0.3730(6)	0.2550(6)	0.3926(5)	0.051(3)	0.040(3)	0.064(4)	0.018(3)	0.006(3)	0.027(3)
Br(1)	2*i*	−0.0417(2)	0.1005(2)	0.2548(2)	0.121(1)	0.150(2)	0.152(2)	0.093(1)	−0.007(1)	0.009(1)
Br(2)	2*i*	0.5899(2)	0.3049(2)	0.8941(1)	0.147(1)	0.168(2)	0.0683(7)	0.046(1)	0.0316(8)	0.0279(9)
Br(3)	2*i*	0.8842(2)	0.3162(2)	0.4662(1)	0.104(1)	0.145(1)	0.0978(9)	0.0153(9)	0.0523(8)	0.0466(9)
C(1)	2*i*	0.572(1)	0.110(1)	0.213(1)	0.101(8)	0.13(1)	0.095(7)	0.065(8)	0.052(6)	0.081(8)
C(2)	2*i*	0.626(2)	0.172(2)	0.158(1)	0.13(1)	0.18(2)	0.13(1)	0.08(1)	0.09(1)	0.11(1)
C(3)	2*i*	0.613(1)	0.278(2)	0.165(1)	0.085(8)	0.16(1)	0.112(9)	0.029(8)	0.035(7)	0.10(1)
C(4)	2*i*	0.553(1)	0.336(1)	0.2296(9)	0.067(6)	0.109(9)	0.083(6)	0.006(6)	0.007(5)	0.071(6)
C(5)	2*i*	0.531(1)	0.456(1)	0.244(1)	0.090(8)	0.092(8)	0.12(1)	0.001(7)	0.002(7)	0.087(8)
C(6)	2*i*	0.462(1)	0.498(1)	0.300(1)	0.107(9)	0.073(7)	0.112(9)	0.028(7)	0.016(7)	0.067(7)
C(7)	2*i*	0.407(1)	0.4314(8)	0.3482(8)	0.087(6)	0.042(4)	0.075(6)	0.019(4)	−0.005(5)	0.031(4)
C(8)	2*i*	0.326(1)	0.4699(9)	0.406(1)	0.107(8)	0.046(5)	0.114(9)	0.036(6)	−0.016(7)	0.026(5)
C(9)	2*i*	0.272(1)	0.4048(9)	0.455(1)	0.079(6)	0.054(5)	0.107(8)	0.039(5)	0.009(6)	0.027(5)
C(10)	2*i*	0.2997(9)	0.2981(8)	0.4473(8)	0.068(5)	0.051(4)	0.081(6)	0.035(4)	0.015(4)	0.027(4)
C(11)	2*i*	0.4231(8)	0.3202(7)	0.3450(7)	0.053(4)	0.044(4)	0.060(4)	0.014(3)	−0.004(3)	0.027(3)
C(12)	2*i*	0.4974(8)	0.2705(8)	0.2819(7)	0.054(4)	0.065(5)	0.063(5)	0.005(4)	−0.007(4)	0.046(4)
C(13)	2*i*	0.8227(7)	0.3100(8)	0.6502(7)	0.053(4)	0.054(4)	0.065(5)	0.024(3)	0.015(3)	0.031(4)
C(14)	2*i*	0.852(1)	0.356(1)	0.765(1)	0.070(7)	0.12(1)	0.14(1)	0.018(7)	0.032(8)	0.08(1)
C(15)	2*i*	0.962(1)	0.455(2)	0.838(1)	0.13(1)	0.17(2)	0.052(6)	0.02(1)	0.007(7)	0.012(8)
C(16)	2*i*	1.048(1)	0.515(2)	0.801(1)	0.081(8)	0.13(1)	0.11(1)	−0.025(8)	0.015(8)	0.02(1)
C(17)	2*i*	1.022(1)	0.476(1)	0.692(1)	0.060(6)	0.101(9)	0.108(9)	−0.009(6)	0.022(6)	0.026(7)
C(18)	2*i*	0.9108(8)	0.3731(9)	0.6174(9)	0.052(5)	0.077(6)	0.094(7)	0.018(4)	0.024(4)	0.040(5)
C(19)	2*i*	0.6936(7)	0.2078(6)	0.5741(7)	0.054(4)	0.034(3)	0.069(4)	0.020(3)	0.025(3)	0.025(3)
C(20)	2*i*	0.0434(9)	−0.086(1)	0.1255(8)	0.064(5)	0.073(6)	0.076(6)	0.007(5)	−0.009(4)	0.037(5)
C(21)	2*i*	−0.057(1)	−0.036(1)	0.133(1)	0.080(7)	0.080(8)	0.14(1)	0.026(6)	−0.023(7)	0.041(8)
C(22)	2*i*	−0.173(1)	−0.080(2)	0.039(1)	0.11(1)	0.13(1)	0.11(1)	0.030(9)	−0.030(8)	0.04(1)
C(23)	2*i*	−0.186(1)	−0.182(2)	−0.057(1)	0.09(1)	0.15(2)	0.11(1)	0.03(1)	−0.027(8)	0.04(1)
C(24)	2*i*	−0.095(1)	−0.241(2)	−0.065(1)	0.13(1)	0.12(1)	0.093(9)	0.02(1)	−0.038(9)	0.010(8)
C(25)	2*i*	0.029(1)	−0.192(1)	0.020(1)	0.11(1)	0.15(1)	0.13(1)	−0.05(1)	−0.069(9)	0.10(1)
C(26)	2*i*	0.1686(8)	−0.0384(8)	0.2239(7)	0.053(4)	0.050(4)	0.061(5)	0.014(3)	0.006(4)	0.024(4)
C(27)	2*i*	0.3426(9)	0.1879(9)	0.7143(8)	0.091(6)	0.079(6)	0.092(6)	0.056(5)	0.055(5)	0.060(5)
C(28)	2*i*	0.419(2)	0.2679(9)	0.832(1)	0.26(2)	0.061(6)	0.16(1)	0.083(9)	0.18(1)	0.063(7)
C(29)	2*i*	0.349(2)	0.321(1)	0.891(1)	0.19(2)	0.11(1)	0.14(1)	0.09(1)	0.12(1)	0.08(1)
C(30)	2*i*	0.218(2)	0.298(2)	0.843(2)	0.18(2)	0.16(2)	0.22(2)	0.10(2)	0.16(2)	0.10(2)
C(31)	2*i*	0.145(2)	0.223(2)	0.731(2)	0.19(2)	0.20(2)	0.27(3)	0.15(2)	0.18(2)	0.18(2)
C(32)	2*i*	0.210(1)	0.166(2)	0.665(1)	0.109(9)	0.15(1)	0.17(1)	0.087(9)	0.09(1)	0.11(1)
C(33)	2*i*	0.4028(7)	0.1202(7)	0.6388(6)	0.056(4)	0.048(4)	0.057(4)	0.028(3)	0.020(3)	0.025(3)

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