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Tetra-μ-acetato-$\kappa^4O:O';\kappa^3O,O':O';\kappa^3O:O,O'$-bis[(acetato-$\kappa^2O,O'$)(1,10-phenanthroline-$\kappa^2N,N'$)-holmium(III)]

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.019 Å
R factor = 0.058
wR factor = 0.133
Data-to-parameter ratio = 14.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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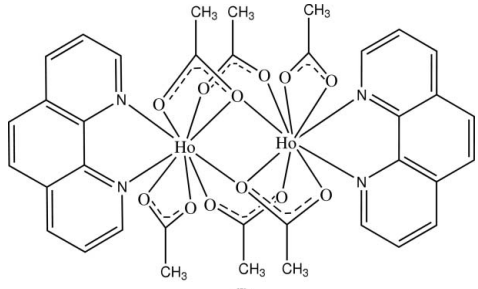
The dimeric title complex, $[Ho_2(CH_3COO)_6(C_{12}H_8N_2)_2]$, lies about a centre of symmetry. Each cation is coordinated by a chelating 1,10-phenanthroline and two chelating acetato ligands, one of which also bridges the two Ho atoms. Two other bridging acetate ligands complete the coordination of the nine-coordinate holmium(III) atoms.

Comment

Dinuclear lanthanide complexes with 1,10-phenanthroline and acetate ligands have received much attention due to their unique properties, such as luminescence and magnetism (Wang *et al.*, 1999). We report here the structure of the dinuclear centrosymmetric title complex, (I), in which the holmium(III) atoms are nine-coordinate (Fig. 1). Panagiotopoulos *et al.* (1995) have reported the structure of a cerium analogue in which the central cerium(III) atoms also adopt this configuration



(I)

Experimental

$Ho(ClO_4)_3{\cdot}6H_2O$ (0.063 g, 0.11 mmol) in MeOH (20 ml) was added to a boiling solution of 1,10-phenanthroline (0.018 g, 0.1 mmol) and sodium acetate (0.025 g, 0.3 mmol) in MeOH (50 ml). After refluxing for 2 h, the mixture was cooled to room temperature. Colourless crystals suitable for X-ray diffraction analysis were obtained in 35% yield by diffusion of Et_2O into the mixture over one week. **Caution!** Although no problems were encountered in this work, perchlorate compounds are potentially explosive. They should be prepared in small amounts and handled with care.

Crystal data

$[Ho_2(C_2H_3O_2)_6(C_{12}H_8N_2)_2]$
M_r = 1044.53
Triclinic, $P\overline{1}$
a = 8.717 (4) Å
b = 8.926 (4) Å
c = 12.914 (6) Å
α = 103.318 (7)°
β = 109.007 (6)°
γ = 98.380 (7)°
V = 897.5 (7) Å^3
Z = 1
D_x = 1.933 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 4.45 mm^{-1}
T = 298 (2) K
Block, colorless
0.05 × 0.03 × 0.03 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.855, T_{max} = 0.873
6456 measured reflections
3449 independent reflections
2749 reflections with $I > 2\sigma(I)$
R_{int} = 0.054
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.133
S = 1.03
3449 reflections
247 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0675P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 2.03 e Å^{-3}
$\Delta\rho_{min}$ = −1.86 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O5	0.93	2.53	3.042 (14)	115
C10—H10···O6	0.93	2.41	3.031 (14)	124
C15—H15A···O2^{i}	0.96	2.46	3.419 (14)	174

Symmetry code: (i) $x, y-1, z$.

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic, and C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms. The highest residual density peak is located 1.07 Å from the Ho atom and the deepest hole is located 1.71 Å from atom C5.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

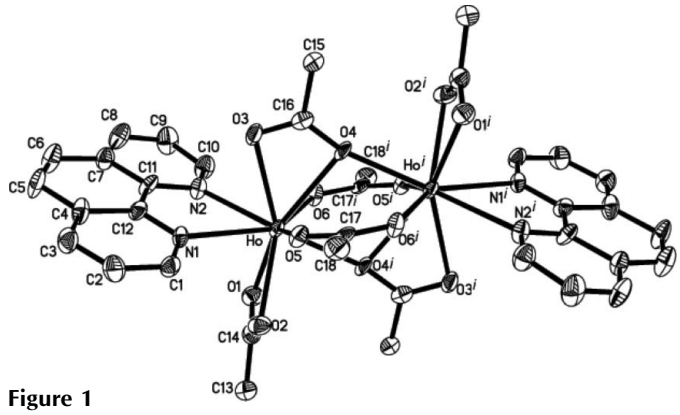


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level. H atoms have been omitted for clarity. [Symmetry code: (i) $2-x, -y, -z$.]

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