Table 2. *Selected geometric parameters* (Å, °)

*M*1 and *M*2 are the centres of the C30—C31 and C34—C35 bonds.

Ir—*M*1	2.005	Ir—*M*2	2.016
Ir—P1	2.410 (2)	Ir—O2	2.724 (6)
Ir—N	2.130 (7)	Ir—C30	2.117 (11)
Ir—C31	2.135 (9)	Ir—C34	2.125 (10)
Ir—C35	2.142 (10)	P1—C1	1.812 (9)
P1—C9	1.848 (8)	P1—C17	1.864 (10)
*M*1—Ir—*M*2	86.4	*M*1—Ir—P1	97.5
*M*1—Ir—O2	103.4	*M*1—Ir—N	170.9
*M*2—Ir—P1	168.4	*M*2—Ir—O2	119.3
*M*2—Ir—N	88.1	P1—Ir—O2	70.6 (1)
P1—Ir—N	86.2 (2)	O2—Ir—N	85.6 (2)
Ir—P1—C1	111.1 (3)	Ir—P1—C9	104.3 (3)
Ir—P1—C17	126.8 (3)	C1—P1—C9	109.4 (4)
C1—P1—C17	97.1 (4)	C9—P1—C17	107.5 (4)

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55996 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1031]

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Structure of $[Yb(OH)(C_{12}H_8N_2)_2(H_2O)_2]_2$-$Cl_4.2C_{12}H_8N_2.CH_3OH$

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Abstract

The title compound, di-μ-hydroxo-bis[diaquabis(1,10-phenanthroline)ytterbium(III)] tetrachloride bis(1,10-phenanthroline) methanol solvate, lies around a crystallographic inversion center with symmetric [2.196 (2) and 2.208 (2) Å] hydroxy bridges. Two terminal water molecules and two bidentate phenanthroline ligands (phen) complete the eight-coordinate square-antiprismatic Yb coordination sphere. Two phen molecules and one methanol molecule are clathrated in the structure.

Comment

The dimeric cationic units, $[Yb(\mu\text{-}OH)(phen)_2(H_2O)_2]_2^{2+}$, are connected *via* hydrogen bonding to the chlorine anions and to the two centrosymmetrically related clathrated phen molecules. The $Yb\cdots Yb^{i}$ separation in the dimer is 3.616 (1) Å. Coordinated and clathrated phen molecules are almost parallel with several contact distances less than 3.6 Å. The molecular packing is dominated by these graphite-type interactions which could be indicative of dipole–dipole interactions among coordinated (in the dimers) and uncoordinated phen molecules. The complex is isostructural with its yttrium analog (Grillone, Benetollo & Bombieri, 1991) and the differences observed in the corresponding bonding parameters are on average 0.028 Å, slightly shorter than expected given the 0.034 Å differences in the effective ionic radii of eight-coordinate Y^{III} and Yb^{III} (Shannon, 1976). The two structures differ slightly in the position of the clathrated methanol molecule.

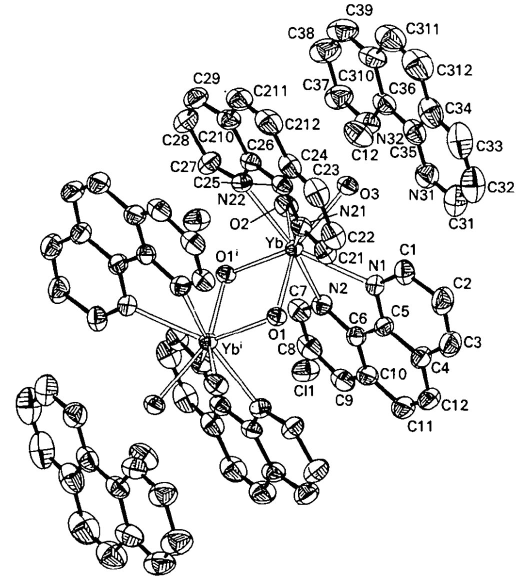

Fig. 1. *ORTEP* illustration (Johnson, 1976) of $[Yb(OH)(phen)_2(H_2O)_2]_2Cl_4.2(phen)$ with 40% probability ellipsoids for thermal motions. H atoms have been omitted for clarity.

Experimental

Crystal data

$[Yb(OH)(C_{12}H_8N_2)_2(H_2O)_2]_2$-$Cl_4.2C_{12}H_8N_2.CH_4O$
$M_r = 1707.27$
Triclinic
$P\bar{1}$
$a = 10.792\ (2)$ Å
$b = 11.369\ (3)$ Å
$c = 13.943\ (3)$ Å
$\alpha = 86.64\ (3)°$
$\beta = 95.24\ (3)°$
$\gamma = 101.91\ (3)°$
$V = 1665.5\ (7)$ Å^3
$Z = 1$

$D_x = 1.702$ Mg m^{-3}
$D_m = 1.69$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10$-$13°$
$\mu = 2.86$ mm^{-1}
$T = 295\ (1)$ K
Prism
0.26 × 0.24 × 0.12 mm
White

Data collection

Philips PW1100 diffractometer
$\theta/2\theta$ scans
Absorption correction: empirical
$T_{min} = 0.70$, $T_{max} = 1.00$
6193 measured reflections
5900 independent reflections
5310 observed reflections $[I > 3\sigma(I)]$

$R_{int} = 0.011$
$\theta_{max} = 26°$
$h = -12 \rightarrow 12$
$k = -13 \rightarrow 13$
$l = 0 \rightarrow 16$
2 standard reflections frequency: 180 min intensity variation: none

Refinement

Refinement on F
Final $R = 0.0158$
$wR = 0.0173$
$S = 1.13$
5302 reflections
554 parameters
All H-atom parameters refined

$w = [\sigma^2(F) + 0.000251(F)^2]^{-1}$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.53$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic thermal parameters* (Å^2)

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Yb	0.026075 (9)	0.106956 (9)	0.094368 (7)	0.02472 (4)
Cl(1)	−0.32625 (7)	−0.16162 (7)	0.16877 (5)	0.0501 (2)
Cl(2)	−0.15426 (8)	0.25267 (8)	0.33868 (6)	0.0597 (3)
O(1)	0.0818 (2)	0.0695 (2)	−0.0465 (1)	0.0298 (6)
O(2)	−0.1447 (2)	0.0742 (2)	0.1892 (2)	0.0431 (7)
O(3)	0.0801 (2)	0.2418 (2)	0.2211 (1)	0.0383 (7)
N(1)	0.0777 (2)	0.3189 (2)	0.0174 (2)	0.0346 (7)
N(2)	−0.1514 (2)	0.1738 (2)	−0.0118 (2)	0.0334 (7)
C(1)	0.1829 (3)	0.3998 (3)	0.0385 (2)	0.0415 (9)
C(2)	0.2253 (3)	0.5008 (3)	−0.0210 (3)	0.054 (1)
C(3)	0.1558 (3)	0.5199 (3)	−0.1045 (3)	0.055 (1)
C(4)	0.0391 (3)	0.4402 (3)	−0.1278 (2)	0.045 (1)
C(5)	0.0042 (3)	0.3420 (2)	−0.0632 (2)	0.036 (1)
C(6)	−0.1194 (3)	0.2650 (2)	−0.0779 (2)	0.035 (1)
C(7)	−0.2695 (3)	0.1107 (3)	−0.0193 (2)	0.041 (1)
C(8)	−0.3620 (3)	0.1354 (3)	−0.0908 (2)	0.048 (1)
C(9)	−0.3298 (3)	0.2235 (3)	−0.1585 (2)	0.048 (1)
C(10)	−0.2044 (3)	0.2927 (3)	−0.1546 (2)	0.044 (1)
C(11)	−0.1626 (4)	0.3893 (3)	−0.2215 (2)	0.054 (1)
C(12)	−0.0465 (4)	0.4589 (3)	−0.2089 (2)	0.055 (1)
N(21)	0.2667 (2)	0.1357 (2)	0.1293 (2)	0.032 (1)
C(21)	0.3513 (3)	0.2027 (3)	0.0746 (2)	0.040 (1)
N(22)	0.1004 (2)	−0.0275 (2)	0.2274 (2)	0.033 (1)
C(22)	0.4820 (3)	0.2286 (3)	0.1015 (3)	0.051 (1)
C(23)	0.5262 (3)	0.1850 (3)	0.1876 (3)	0.054 (1)
C(24)	0.4409 (3)	0.1103 (3)	0.2457 (2)	0.044 (1)
C(25)	0.3112 (2)	0.0855 (2)	0.2127 (2)	0.034 (1)
C(26)	0.2233 (2)	−0.0005 (2)	0.2651 (2)	0.033 (1)
C(27)	0.0205 (3)	−0.1146 (3)	0.2717 (2)	0.042 (1)
C(28)	0.0569 (3)	−0.1766 (3)	0.3562 (2)	0.052 (1)
C(29)	0.1795 (3)	−0.1468 (3)	0.3960 (2)	0.050 (1)
C(210)	0.2664 (3)	−0.0580 (3)	0.3500 (2)	0.043 (1)
C(211)	0.3981 (3)	−0.0241 (3)	0.3842 (2)	0.054 (1)
C(212)	0.4801 (3)	0.0542 (3)	0.3351 (3)	0.055 (1)
N(32)	−0.7628 (2)	0.2440 (2)	0.3893 (2)	0.047 (1)
N(31)	−0.6123 (3)	0.4077 (2)	0.2747 (2)	0.053 (1)
C(31)	−0.5357 (4)	0.4868 (3)	0.2219 (3)	0.065 (1)
C(32)	−0.4081 (4)	0.5349 (4)	0.2484 (4)	0.078 (2)
C(33)	−0.3590 (4)	0.5016 (4)	0.3352 (4)	0.077 (2)
C(34)	−0.4351 (3)	0.4203 (3)	0.3969 (3)	0.062 (1)
C(35)	−0.5622 (3)	0.3755 (3)	0.3622 (2)	0.046 (1)
C(36)	−0.6427 (3)	0.2913 (3)	0.4235 (2)	0.045 (1)
C(37)	−0.8344 (4)	0.1662 (4)	0.4460 (3)	0.060 (1)
C(38)	−0.7930 (4)	0.1296 (4)	0.5382 (3)	0.069 (2)
C(39)	−0.6728 (4)	0.1785 (4)	0.5742 (3)	0.069 (2)
C(310)	−0.5938 (3)	0.2604 (3)	0.5162 (2)	0.056 (1)
C(311)	−0.4645 (4)	0.3120 (5)	0.5482 (3)	0.073 (2)
C(312)	−0.3903 (4)	0.3861 (4)	0.4911 (4)	0.079 (2)
O†	0.0000	0.5000	0.5000	0.172 (4)
C†	0.069 (1)	0.422 (1)	0.4816 (8)	0.099 (3)

† Occupancy factor of 0.5 and U_{iso} value.

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Yb—O(1)	2.196 (2)	Yb—N(2)	2.528 (2)
Yb—O(1ⁱ)	2.208 (2)	Yb—N(21)	2.555 (2)
Yb—O(2)	2.321 (2)	Yb—N(22)	2.514 (2)
Yb—O(3)	2.361 (2)	N—C (average)	1.342 (4)
Yb—N(1)	2.554 (2)	C—C (average)	1.397 (5)
Yb—O(1)—Ybⁱ	110.4 (1)	O(1)—Yb—O(1ⁱ)	69.7 (1)

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

The title complex was isolated in a similar manner to the yttrium analog (Grillone *et al.*, 1991) by reacting $YbCl_3.6H_2O$ with a MeOH solution of (1,10-phenanthroline).H_2O in a molar ratio of about 1:6. IR spectra reveal two reaction products, depending on the experimental conditions. Good prismatic crystals of the title compound have only been obtained in about 50% yield from a MeOH/Me_2CO mixture.

The clathrated methanol molecule (one per dimer) with the O atom at the inversion center $(0,\frac{1}{2},\frac{1}{2})$ is statistically disordered about this position.

Calculations were carried out using *SHELX76* (Sheldrick, 1976).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71042 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: NA1028]

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Structure of Bis(hydronium) Bis(pyrazine-2,3-dicarboxylato)manganate, a Polymeric Mn^{II} Complex

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Abstract

The structure of the title compound consists of centrosymmetric anionic units, $[Mn(C_6H_2N_2O_4)_2]^{2-}$, which are linked by hydrogen bonds to the hydronium cations. The Mn atom is in a six-coordinate N_2O_4 octahedral environment. One carboxylato group acts as a bridge between the molecular units and gives the polymeric structure.

Comment

Manganese(II) forms a variety of compounds with dicarboxylates (Chiswell, McKenzie & Lindoy, 1987), or carboxylates having other functional groups. Pyrazinedicarboxylate is a potential tetradentate ligand with different donor sites comprised of the O atoms of the dicarboxylic acid and the N atoms of the aromatic ring. Complexes with some metallic cations have been prepared. Their insolubility in both polar and non-polar solvents was indicative of polymeric structure, however, single-crystal X-ray structure analysis was not possible (Allan, Paton, Turvey, Bowley & Gerrard, 1988). In this work the structure of a polymeric Mn^{II} complex of pyrazinedicarboxylate is reported which is the first example involving this type of ligand.

The Mn atom sits on a crystallographic centre of symmetry in a distorted octahedral arrangement with each of the pyrazinedicarboxylate ligands acting as a dianionic tridentate NO_2 donor. Mn^{II} is bound to two bidentate chelating groups [O(1) and N(2)] of two pyrazinedicarboxylate ligands and to two monodentate carboxylato groups [O(3)] from two different molecular units, resulting in the polymeric structure. The hydronium counterion acts as a bridge between

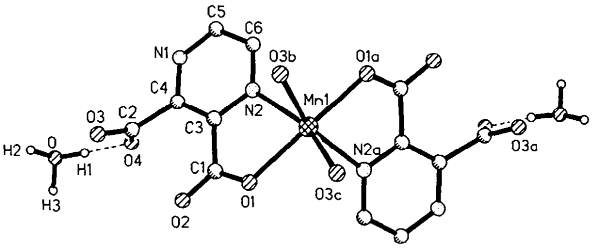

Fig. 1. A perspective view of the molecule with the atom-numbering scheme.

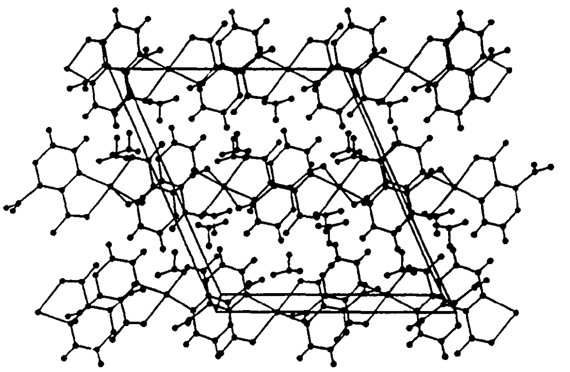

Fig. 2. Projection of the unit cell viewed along [010].