

Fig. 2. Packing in  $1:\frac{1}{2}$  (triphenylarsine)diiodine toluene solvate (III). Two other identical columns generated by centring operations,  $\frac{2}{3}$ ,  $\frac{1}{3}$ ,  $\frac{1}{3}$  and  $\frac{1}{3}$ ,  $\frac{2}{3}$ ,  $\frac{2}{3}$  complete the cell contents. Half the disordered toluene molecules have been omitted for clarity.

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## Structure of Pentaqua(L-threonine)holmium(III) Trichloride

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**Abstract.**  $[\text{Ho}(\text{H}_2\text{O})_5(\text{C}_4\text{H}_9\text{NO}_3)]\text{Cl}_3$ ,  $M_r = 480.5$ , orthorhombic,  $P2_12_12_1$ ,  $a = 10.508$  (3),  $b = 7.395$  (2),  $c = 18.708$  (4) Å,  $V = 1453.7$  Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 2.19$ ,  $D_x = 2.20$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 6.22$  mm<sup>-1</sup>,  $F(000) = 928$ ,  $T = 298$  (2) K. Final  $R = 0.040$  for 2700 observed reflections. The structure is that of a linear polymer. The threonine molecule coordinates to Ho *via* chelation through the hydroxy O and one of the carboxyl O atoms. The second carboxyl O atom is coordinated to Ho of a symmetry-related molecule to form polymeric chains along *a*. Ho is eight-coordinate with a coordination polyhedron in the form of a distorted square antiprism.

**Introduction.** The complexes of hydroxycarboxylic acids with lanthanide ions were investigated by Grenthe

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(1972, and references therein). The results of these investigations show that hydroxycarboxylate ions coordinate to metal ions as chelate ligands *via* the hydroxy O and one of the carboxyl O atoms. In this paper we have determined the structure of the title compound, in order to elucidate how the coordination mode might change if the hydroxycarboxylic acid contained an additional amine group.

**Experimental.** Crystals of the title compound were obtained from an aqueous solution of  $\text{HoCl}_3$  and L-threonine; density by flotation in bromoform/ethylene bromide. Intensity data with  $2\theta_{\text{max}} = 65.0^\circ$  (Mo  $K\alpha$  radiation) collected from crystal of size  $0.20 \times 0.25 \times 0.40$  mm, Syntex  $P2_1$  diffractometer,  $\theta:2\theta$  scan with scan rate  $3.0\text{--}29.3^\circ \text{min}^{-1}$ , systematic absences  $h00$

$h = 2n + 1$ ,  $0k0$   $k = 2n + 1$ ,  $00l$   $l = 2n + 1$ , cell parameters determined by least squares from 15 reflections in the range  $20 < 2\theta < 30^\circ$ ; two standards with intensity variations  $\pm 3\%$  every 50 reflections. 3001 reflections measured with 2700 reflections with  $I > 4.0\sigma(I)$  used for structure determination; index range  $h-15$  to  $15$ ,  $k-11$  to  $11$ ,  $l$  0 to 28. Intensities corrected for absorption with the program *DIFABS* (Walker & Stuart, 1983). Structure solved *via* Patterson and difference-Fourier techniques. Full-matrix least-squares refinement minimizing  $\sum w(|F_o| - |F_c|)^2$ ,  $w = 1/\sigma^2(F)$ ;

Table 1. Positional parameters and equivalent isotropic temperature factors ( $\text{\AA}^2$ ) for the non-H atoms with e.s.d.'s in parentheses

	$B_{eq} = \frac{1}{3} \sum_i B_{ii}$			
	x	y	z	$B_{eq}$
Ho	0.02976 (4)	0.25651 (7)	0.58300 (2)	0.77 (2)
Cl(1)	0.2084 (3)	0.2528 (6)	-0.0201 (2)	2.3 (2)
Cl(2)	0.3304 (3)	0.2225 (5)	0.7871 (2)	2.1 (2)
Cl(3)	0.3143 (4)	0.2627 (6)	0.2177 (2)	2.9 (2)
O(1)	-0.1346 (7)	0.2153 (11)	0.4996 (4)	1.2 (5)
O(2)	-0.2581 (8)	0.1538 (13)	0.4090 (4)	1.4 (5)
O(3)	0.1239 (7)	0.2039 (12)	0.4672 (4)	1.4 (5)
O(4)	0.0775 (8)	0.2389 (17)	0.7045 (4)	2.3 (6)
O(5)	-0.1300 (14)	0.4398 (15)	0.6376 (6)	2.5 (9)
O(6)	-0.1208 (13)	0.0473 (13)	0.6352 (6)	2.3 (8)
O(7)	0.0437 (10)	0.5502 (12)	0.5352 (5)	2.2 (6)
O(8)	0.1317 (12)	-0.0247 (13)	0.5859 (5)	2.8 (8)
N	-0.0778 (10)	-0.0040 (14)	0.3312 (5)	1.4 (6)
C(1)	-0.1554 (10)	0.1496 (15)	0.4392 (5)	0.9 (6)
C(2)	-0.0434 (10)	0.0607 (15)	0.4035 (5)	1.0 (6)
C(3)	0.0710 (11)	0.1924 (17)	0.3975 (6)	1.4 (7)
C(4)	0.0340 (17)	0.3797 (20)	0.3695 (7)	2.7 (10)

Table 2. Selected geometry

Distances in  $\text{\AA}$  and angles in  $^\circ$  with e.s.d.'s in parentheses. Symmetry code: (i)  $\frac{1}{2} + x$ ,  $\frac{1}{2} - y$ ,  $1 - z$ .

(a) Bonded Ho—O and threonine distances

Ho—O(1)	2.347 (7)	Ho—O(2')	2.331 (8)
Ho—O(3)	2.414 (8)	Ho—O(4)	2.331 (7)
Ho—O(5)	2.387 (13)	Ho—O(6)	2.419 (12)
Ho—O(7)	2.354 (9)	Ho—O(8)	2.340 (10)
C(1)—O(1)	1.25 (1)	C(1)—O(2)	1.22 (1)
C(1)—C(2)	1.51 (2)	C(2)—C(3)	1.55 (2)
C(3)—C(4)	1.53 (2)	C(3)—O(3)	1.42 (1)
C(2)—N	1.48 (1)		

(b) Non-bonded O...O distances

O(5)...O(1)	3.07 (1)	O(5)...O(7)	2.77 (2)
O(7)...O(1)	3.18 (1)	O(4)...O(8)	3.01 (1)
O(4)...O(2)	2.85 (1)	O(2)...O(8)	2.99 (1)
O(5)...O(4)	2.92 (2)	O(1)...O(8)	3.69 (1)
O(7)...O(2)	2.78 (1)	O(6)...O(5)	2.90 (2)
O(6)...O(4)	2.83 (2)	O(6)...O(8)	2.86 (2)
O(6)...O(1)	2.83 (1)	O(3)...O(1)	2.78 (1)
O(3)...O(7)	2.98 (1)	O(3)...O(2')	2.83 (1)
O(3)...O(8)	2.79 (1)	O(6)...O(3)	4.22 (1)

(c) Valence angles

O(8)—Ho—O(1)	103.7 (3)	O(3)—Ho—O(6)	121.8 (3)
O(4)—Ho—O(7)	114.1 (3)	O(2)—Ho—O(5)	118.9 (4)
O(1)—C(1)—O(2)	124.4 (10)	O(1)—C(1)—C(2)	115.8 (9)
O(2)—C(1)—C(2)	119.8 (10)	C(1)—C(2)—N	110.9 (9)
C(1)—C(2)—H(1)	108	N—C(2)—C(3)	109.1 (9)
C(2)—C(3)—C(4)	113.3 (10)	C(2)—C(3)—O(3)	106.1 (9)
C(4)—C(3)—O(3)	111.1 (10)		

anisotropic thermal factors for non-H atoms, common isotropic temperature factor for H atoms; final  $R = 0.040$ ,  $wR = 0.063$ ,  $S = 5.092$ , max.  $\Delta/\sigma = 0.02$ ; max.  $\Delta\rho$  within  $\pm 0.40 \text{ e \AA}^{-3}$ ; all calculations performed on a Nova 1200 computer using the *XTL/XTLE* programs (Syntex, 1976). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Final atomic parameters are given in Table 1.\*

**Discussion.** The selected distances and angles with e.s.d.'s are given in Table 2. The crystal structure is illustrated in Fig. 1. The threonine molecule coordinates to the Ho ion as a chelate ligand *via* the hydroxyl O and one of the carboxyl O atoms. The second O atom of the carboxyl group coordinates to a symmetry-related Ho atom with  $\text{Ho}\cdots\text{Ho} = 6.104 (2) \text{ \AA}$ . Thus the coordination modes of the carboxyl group in the threonine complex are similar to those of hydroxyacetate ions (Grenthe, 1972) with lanthanide ions. The

\* Lists of structure factors, anisotropic thermal parameters, H-atom positions, torsion angles and details of least-squares planes and of hydrogen bonds have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44239 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

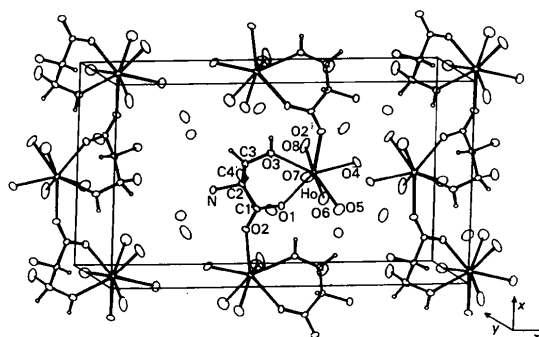


Fig. 1. The crystal structure with atomic numbering.

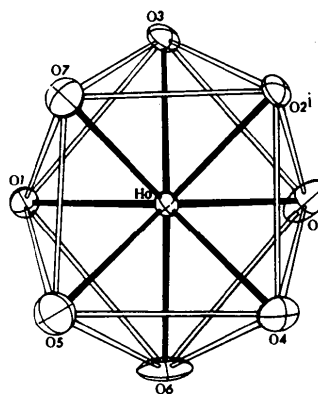


Fig. 2. The holmium coordination polyhedron.

Table 3. *Shape characteristics for coordination polyhedron of the title compound in comparison with hard sphere model (HSM) and most favourable polyhedron (MFP)*

$\Delta_{\text{SAP}}$	$\Delta_{\text{BCTP}}$	$\sigma$				$\varphi$	$\theta$	$l/s$	$t$	$v_1$	$h_1$	$h_3$	$v_2$	$c_2$	$c_1$	$h_2$
0.023	0.073	4.6	16.8	55.4	56.8	29.9	57.3	1.05	4.22	3.69	3.09	3.02	2.85	2.89	2.82	2.81
HSM <sub>SAP</sub>		0.0	0.0	52.4	52.4	24.5	59.2	1.10								
HSM <sub>BCTP</sub>		0.0	21.8	48.2	48.2	14.1										
MFP <sub>BCTP</sub>									1.78	1.50	1.36	1.36	1.25	1.21	1.18	1.13

The  $\Delta$  values (Å) are calculated by the equation  $\Delta = \sum_{i=1}^8 d_i^2/8$ , where  $d_i$  is distance between comparable points in the observed and ideal polyhedron. SAP: square antiprism; BCTP: bicapped trigonal prism. ( $\sigma$ ,  $\varphi$  and  $\theta$  are in °;  $t$ ,  $v_1$ ,  $h_1$ ,  $h_3$ ,  $v_2$ ,  $c_2$ ,  $c_1$  and  $h_2$  are in Å.)

difference between the structure of the present threonine compound and the hydroxyacetate compounds is that the former consists of one-dimensional polymeric chains, while the latter consist of three-dimensional polymeric networks.

The Ho ion is eight-coordinated. The coordination polyhedron (Fig. 2) is so distorted that it can be described as intermediate between a square antiprism and a bicapped trigonal prism. The 'square' faces of the antiprism are made up of O(1), O(3), O(8), O(6) and O(2<sup>1</sup>), O(4), O(5), O(7). The dihedral angle between the 'square' faces is 4.9 (8)°. The  $\Delta$  value, defined as  $\sum d_i^2/8$  for  $i = 1-8$ , is 0.023 Å. Here  $d_i$  is the distance between the observed position of the  $i$ th atom (vertex) and the relevant one in the ideal, least-squares-fitted polyhedron (Drew, 1977). The triangular faces of the trigonal prism comprise the atoms O(1), O(5), O(7) and O(2<sup>1</sup>), O(4), O(8). The dihedral angle between these faces is 18.1 (9)°. The hydroxyl oxygen O(3) and water oxygen O(6) form the caps of the prism. In this case  $\Delta = 0.073$  Å. The other shape characteristics of the coordination polyhedron calculated in comparison with ideal polyhedra are given in Table 3.

The bond distances and angles of L-threonine in the present structure do not show any significant differences in comparison with those in free (zwitterionic)

L-threonine (Shoemaker, Donohue, Schomaker & Corey, 1950). The torsion angles Ho—O(1)—C(1)—C(2) and Ho—O(2)—C(1)—C(2) are 1.3 (12) and -141.5 (8)°, respectively. Other torsion angles have been deposited.\* Each threonine—Ho chelate forms a six-membered ring which has a twist-boat conformation [ $Q = 0.659$  (9),  $q_2 = 0.623$  (9) Å,  $\theta = 71.0$  (8),  $\varphi = 202.5$  (9)°]. The crystal structure is stabilized by hydrogen bonds of the type O—H...O, O—H...Cl and N—H...Cl.

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\* See deposition footnote.

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## Carbonatobis(triphenylphosphine)platinum(II) Tetrahydrofuran Solvate

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**Abstract.** [Pt(CO<sub>3</sub>){P(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>}]<sub>2</sub>.C<sub>4</sub>H<sub>8</sub>O,  $M_r = 851.8$ , triclinic,  $P\bar{1}$ ,  $a = 10.666$  (2),  $b = 12.359$  (2),  $c = 15.525$  (4) Å,  $\alpha = 83.73$  (2),  $\beta = 70.48$  (3),  $\gamma = 69.76$  (2)°,  $V = 1810$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.56$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 40.4$  cm<sup>-1</sup>,  $F(000) = 848$ ,  $T = 298$  K,  $R = 0.029$  for 5493 reflections with  $I >$

$3\sigma(I)$ . Structure is essentially a redetermination of that originally published by Cariati, Mason, Robertson & Ugo [*J. Chem. Soc. Chem. Commun.* (1967), p. 408] but with tetrahydrofuran instead of benzene solvent in the lattice. Features not observed or commented upon in the original include the presence of