Fig. 1. The molecular structure with the atom labels.

Table 3. *Intermolecular contacts* (Å)

F(1)···F(6)ᴵ	2·77 (1)	C(8)···F(4)ᵛ	3·12 (1)
O(3)···F(12)ᴵᴵ	2·94 (1)	O(1)···F(9)ᵛᴵ	3·17 (1)
O(4)···F(7)ᴵᴵᴵ	3·06 (1)	O(4)···F(3)ᵛᴵᴵ	3·17 (1)
O(2)···F(9)ᴵⱽ	3·07 (1)	F(3)···F(8)ᴵⱽ	3·18 (1)
F(7)···F(8)ᴵⱽ	3·10 (1)	F(5)···F(10)ᴵᴵᴵ	3·18 (1)
O(3)···F(4)ᵛ	3·10 (1)	F(3)···F(7)ᴵⱽ	3·19 (1)

The superscripts refer to the following transformations of the atomic coordinates:

(I)	$1-x$, $-\frac{1}{2}+y$, $1\frac{1}{2}-z$	(V)	$-x$, $-\frac{1}{2}+y$, $1\frac{1}{2}-z$
(II)	$-\frac{1}{2}+x$, $-\frac{1}{2}-y$, $1-z$	(VI)	x, $-1+y$, z
(III)	$-\frac{1}{2}+x$, $\frac{1}{2}-y$, $1-z$	(VII)	$-1+x$, y, z.
(IV)	$\frac{1}{2}+x$, $\frac{1}{2}-y$, $1-z$		

Å in Co₃(CO)₈(CMe)(PPh₃) (Brice, Penfold, Robinson & Taylor, 1970), 1·79–1·83 Å in Co₂(CO)₆(C₈H₁₂-As₂F₄) (Harrison & Trotter, 1971) and 1·75–1·78 Å in Co₃(CO)₇(C₁₀H₁₅As₂F₄) (Einstein & Jones, 1972), and Co–C distances of 1·99 Å in Co(C₂H₅)(C₁₆H₁₄N₂O₂) (Calligaris, Minichelli, Nardin & Randaccio, 1971),

2·04–2·06 Å in Co{(B₉C₂H₁₀)₂S₂CH} (Churchill & Gold, 1971) and 2·02–2·09 Å in Co(C₈H₁₃)(C₈H₁₂) (Koda, Takenaka & Watanabé, 1971). The Co–Co separation of 2·552 Å in (I) is slightly longer than the values of 2·49–2·51 in Co₃(CO)₈(CMe)(PPh₃), 2·48 in Co₂(CO)₆(C₈H₁₂As₂F₄) and 2·44–2·48 Å in Co₃(CO)₇(C₁₀H₁₅As₂F₄).

The F–C–F angles in the organic ligand (mean 107·1°) are distinctly smaller than the C–C–F angles (mean 111·7°). This result is in accord with the relative electronegativities of C and F.

References

BRICE, M. D., PENFOLD, B. R., ROBINSON, W. T. & TAYLOR, S. R. (1970). *Inorg. Chem.* **9**, 362–367.
CALLIGARIS, M., MINICHELLI, D., NARDIN, G. & RANDACCIO, L. (1971). *J. Chem. Soc. A*, pp. 2720–2724.
CHURCHILL, M. R. & GOLD, K. (1971). *Inorg. Chem.* **10**, 1928–1933.
DAVIDSON, J. L. & SHARP, D. W. A. (1975). *J. Chem. Soc. Dalton Trans.* pp. 2283–2287.
EINSTEIN, F. W. B. & JONES, R. D. G. (1972). *Inorg. Chem.* **11**, 395–400.
ENEMARK, J. H. & FELTHAM, R. D. (1972). *J. Chem. Soc. Dalton Trans.* pp. 718–722.
HARRISON, W. & TROTTER, J. (1971). *J. Chem. Soc. A*, pp. 1607–1609.
KODA, S., TAKENAKA, A. & WATANABÉ, T. (1971). *Bull. Chem. Soc. Jpn*, **44**, 653–658.
LEWIS, D. F., LIPPARD, S. J. & ZUBIETA, J. A. (1972). *J. Am. Chem. Soc.* **94**, 1563–1575.
PORTA, P., TARANTELLI, T., GASTALDI, L. & FURLANI, C. (1971). *Inorg. Chim. Acta*, **5**, 616–622.
STEWART, J. M., KUNDELL, F. A. & BALDWIN, J. C. (1970). The XRAY 70 system. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Holmium Triacetate Tetrahydrate

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Abstract. Ho(C₂H₃O₂)₃.4H₂O, triclinic, P$\bar{1}$, $Z = 2$, $a = 9.246$ (3), $b = 9.361$ (3), $c = 10.588$ (4) Å, $\alpha = 90.30$ (3), $\beta = 114.93$ (2), $\gamma = 56.94$ (2)°, $V = 665.3$ (3) Å³, $D_c = 2.07$ Mg m⁻³, μ(Mo $K\alpha$) = 6.20 mm⁻¹; Nb-filtered Mo $K\alpha$ radiation; final $R(F) = 0.053$. The basic structural element consists of a centrosymmetric dimer with formula Ho₂(C₂H₃O₂)₆-(H₂O)₄. The bonding between dimers consists of hydrogen bonds. The coordination of Ho can be described by a distorted three-vertex trigonal prism with a coordination number of 9. The Ho–O distances range between 2.327 (7) and 2.561 (8) Å. The shortest

Ho—Ho distance is 4·159 (2) Å. The title compound is paramagnetic between 4·2 and 300 K.

Introduction. Crystals of $Ho(C_2H_3O_2)_3.4H_2O$ were obtained from an aqueous solution of Ho_2O_3 and acetic acid. A crystal 0·07 × 0·15 × 0·28 mm was selected. The lattice constants were determined from 12 reflections centred on a Syntex $P2_1$ diffractometer. They differ slightly from the values given by Vadura & Kvapil (1971).

One hemisphere of reflections was collected on a Syntex $P2_1$ diffractometer up to $\sin\theta/\lambda = 0.65$ Å^{-1} with a $\theta/2\theta$ scan, resulting in 3072 independent observations. Three standard reflections observed after every 45 reflections showed a gradual decrease of about 30% during the data collection, possibly due to a drift in the high voltage of the detector and radiation damage to the crystal. This effect was corrected by rescaling the reflections with respect to the standards. The data were corrected for absorption; the transmission factor ranged from 0·39 to 0·67. A weighting scheme $w(I) = [\sigma^2(I)_{counting} + (0.03I)^2]^{-1}$ was used in the refinement.

The Ho atom was located by the Patterson method. A subsequent Fourier synthesis yielded the C and O atoms. A difference synthesis after refinement with anisotropic thermal parameters yielded the positions of the hydrate H atoms. They were included in the refinement with isotropic thermal parameters fixed at $B = 4$ Å^2. The positions of the methyl H atoms were less clear, possibly due to the larger thermal motion of these

groups. Scattering factors were from *International Tables for X-ray Crystallography* (1974), except for H (Stewart, Davidson & Simpson, 1965). For Ho the anomalous-dispersion factors of Cromer & Liberman (1970) were applied ($f' = -0.666$, $f'' = 4.678$).

The final $R(F) = 5.3\%$ and $R_w(F) = 5.0\%$. The calculations were carried out with the XRAY system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972) on the Univac 1108 computer of the University of Frankfurt. The positional parameters of the atoms are reported in Table 1, bond distances and angles in Table 2. A stereoscopic view of the structure is given in Fig. 1.*

Discussion. The rare-earth acetates crystallize either as anhydrous (Karraker, 1969) or as hydrated salts. The lighter rare earths form mono-, sesqui- and dihydrated

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Table 1. *Positional parameters* ($\times 10^4$, *for* H $\times 10^3$), *with e.s.d.'s in parentheses*

	x	y	z
Ho	1848 (1)	4112 (1)	4103 (1)
C(1)	1752 (12)	5652 (10)	7130 (9)
C(2)	3596 (14)	5572 (14)	7825 (12)
C(3)	−450 (13)	2318 (11)	6679 (10)
C(4)	303 (17)	402 (12)	7053 (14)
C(5)	3086 (13)	2843 (12)	2083 (9)
C(6)	3625 (16)	2220 (14)	910 (11)
O(1)	1280 (8)	5201 (7)	5974 (6)
O(2)	640 (9)	6174 (8)	7675 (7)
O(3)	739 (8)	2725 (7)	7084 (6)
O(4)	−2329 (9)	3557 (7)	5924 (7)
O(5)	3637 (9)	1724 (7)	3167 (7)
O(6)	2022 (10)	4485 (7)	1958 (7)
O(7)	2854 (10)	1586 (8)	5636 (8)
O(8)	4826 (10)	7226 (10)	4200 (8)
O(9)	823 (12)	7408 (8)	55 (8)
O(10)	7255 (12)	1044 (10)	8711 (8)
H(10)	273 (16)	169 (13)	628 (11)
H(11)	384 (15)	63 (12)	616 (10)
H(12)	429 (17)	686 (16)	410 (13)
H(13)	432 (15)	784 (12)	335 (10)
H(14)	145 (16)	636 (13)	66 (11)
H(15)	94 (15)	708 (13)	−60 (10)
H(16)	820 (17)	4 (13)	897 (11)
H(17)	822 (15)	137 (12)	915 (10)

Table 2. *Bond lengths* (Å) *and angles* (°), *with e.s.d.'s in parentheses*

C(1)—C(2)	1·50 (2)	O(9)—H(14)	0·88 (11)
C(1)—O(1)	1·27 (1)	O(9)—H(15)	0·78 (12)
C(1)—O(2)	1·26 (1)	O(10)—H(16)	0·79 (9)
C(3)—C(4)	1·51 (2)	O(10)—H(17)	1·02 (15)
C(3)—O(3)	1·26 (2)	Ho—O(8)	2·327 (7)
C(3)—O(4)	1·28 (1)	Ho—O(7)	2·337 (8)
C(5)—C(6)	1·53 (2)	Ho—O(1)	2·348 (7)
C(5)—O(5)	1·27 (1)	Ho—O(6)	2·384 (9)
C(5)—O(6)	1·26 (1)	Ho—O(3)	2·418 (5)
O(7)—H(10)	0·74 (13)	Ho—O(2)	2·434 (8)
O(7)—H(11)	0·79 (7)	Ho—O(4)	2·455 (9)
O(8)—H(12)	0·72 (18)	Ho—O(5)	2·457 (7)
O(8)—H(13)	0·85 (11)	Ho—O(1)'	2·561 (8)
C(2)—C(1)—O(1)	121 (1)	C(6)—C(5)—O(6)	120 (1)
C(2)—C(1)—O(2)	121 (1)	O(5)—C(5)—O(6)	119 (1)
O(1)—C(1)—O(2)	119 (1)	H(10)—O(7)—H(11)	85 (11)
C(4)—C(3)—O(3)	121 (1)	H(12)—O(8)—H(13)	104 (13)
C(4)—C(3)—O(4)	120 (1)	H(14)—O(9)—H(15)	98 (11)
O(3)—C(3)—O(4)	119 (1)	H(16)—O(10)—H(17)	88 (13)
C(6)—C(5)—O(5)	121 (1)		
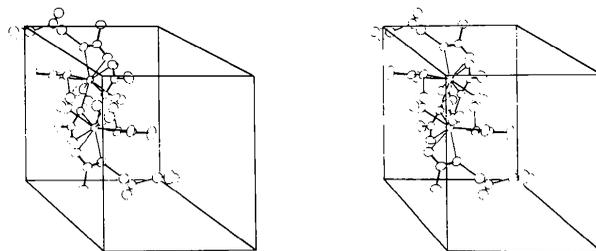
Fig. 1. A stereoscopic view of the structure.

Table 3. *Hydrogen bonds*

O—H···O	O···O	H···O	∠O—H···O
O(7)—H(10)···O(3)	2·748 (12) Å	2·10 (15) Å	147 (10)°
O(7)—H(11)···O(5)	2·709 (7)	1·95 (8)	160 (13)
O(8)—H(12)···O(4)	2·734 (15)	2·05 (19)	161 (14)
O(8)—H(13)···O(10)	2·734 (11)	1·91 (9)	163 (15)
O(9)—H(14)···O(6)	2·764 (12)	1·91 (14)	162 (13)
O(9)—H(15)···O(2)	2·745 (11)	1·98 (11)	164 (16)
O(10)—H(16)···O(9)	2·888 (8)	2·11 (8)	169 (12)
O(10)—H(17)···O(9)	2·792 (16)	1·81 (14)	160 (9)

acetates while the tetrahydrate is the predominant form for Sm to Lu. The tetrahydrates are isostructural and crystallize in space group $P\bar{1}$ (Vadura & Kvapil, 1971). We determined the structure of $Ho(C_2H_3O_2)_3.4H_2O$ whose structural details compare closely with those of the isostructural Er compound (Aslanov, Abdul'minev, Porai-Koshits & Ivanov, 1972).

The basic structural element consists of a centrosymmetric dimer formed by two $Ho(Ac)_3$ units and four water molecules. The link within the dimer is formed by O(1) which belongs to the coordination sphere of two Ho and by the water molecules O(7) and O(8). Two independent carboxyl groups are in the coordination sphere of only one Ho, and are bidentate; the other [with O(1)] is tridentate bridge-cyclic. The dimers are interconnected by water molecules O(9) and O(10). All hydrate H atoms are involved in medium to weak hydrogen bonds (Table 3).

Ho has a ninefold coordination of O atoms, seven from carboxyl groups, and two from water molecules. The Ho—O bonds to the water molecules are slightly shorter than those to the carboxyl groups. The C—O distances of 1·26 (2) to 1·28 (2) Å are normal for carboxyl groups. They do not confirm the results of

Aslanov *et al.* (1972) who report large differences within one carboxyl group [1·19 (4) and 1·35 (4) Å]. The O—C—O angles of 119 (1)° are rather small but not unusual. The shortest Ho—Ho distance within the dimer is 4·159 (2) Å. A superexchange *via* O(1) is possible between these two atoms, and may lead to possible magnetic ordering of the rare earths. Further distances of 6·222, 6·445 and 7·671 Å may involve supersuperexchange *via* carboxyl groups and water molecules. The measurement of the magnetic susceptibility, however, revealed paramagnetic behaviour between 4·2 and 300 K.

References

Aslanov, L. A., Abdul'minev, I. K., Porai-Koshits, M. A. & Ivanov, V. I. (1972). *Dokl. Akad. Nauk SSSR*, **205**, 343–345.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

Karraker, D. G. (1969). *J. Inorg. Nucl. Chem.* **31**, 2815–2832.

Stewart, J. M., Kruger, G. J., Ammon, H. L., Dickinson, C. & Hall, S. R. (1972). The XRAY system – version of June 1972. Tech. Rep. TR-192. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Vadura, R. & Kvapil, J. (1971). *Mater. Res. Bull.* **6**, 865–875.

Bis(μ-trifluoroacetato-O,O′)-bis[dimethyltin(IV)]

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Abstract. $Sn_2(CH_3)_4(C_2F_3O_2)_2$, $C_8H_{12}F_6O_4Sn_2$, $M_r = 523\cdot55$, monoclinic, space group $C2/m$, $a = 14\cdot887$ (5), $b = 8\cdot223$ (5), $c = 7\cdot518$ (3) Å, $\beta = 118\cdot84$ (2)°, $D_x = 2\cdot15$ Mg m^{-3}, $Z = 2$. The structure was refined from 1027 diffractometer-measured X-ray reflections to $R_w = 0\cdot042$. The dimer is isostructural with $Sn_2(CH_3)_4(C_2H_2ClO_2)_2$, having crystallographic $2/m$ symmetry with Sn—Sn = 2·707 (1), Sn—O(av) = 2·332 (3), C—O(av) = 1·23 (1), Sn—C = 2·10 (1) and C—C = 1·536 (9) Å. The fluorine atoms are disordered. The molecule possesses almost *mmm* symmetry, deviations from this being entirely attributable to a weak intermolecular C—H···O interaction.

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