The distortions are shown to be more significant for the endocyclic angles than for the bond distances. Norrestam & Schepper have classified the COOH and NO_2 substituents into two groups on the basis of the torsion angles. The considerable disparity in deformation parameters between the groups means that one cannot expect too close an agreement between observed and calculated values for all combinations of torsion angles of these substituents. Although the present compound contains the benzene derivatives as ionized ligands, a comparison between observed and calculated angles can still be meaningful. It should be noted that the e.s.d.'s of the angles of the ligands are about twice and those of the *o*-nitrobenzoic acid about three times the e.s.d.'s of the angles for compounds accepted by Norrestam & Schepper into their analysis.

The *o*-nitrobenzoic acid and one of the ligands belong to the same group on the basis of the torsion angles of the substituents: in each the COOH (COO^-) substituent is coplanar and the NO_2 substituent orthogonal (Table 3). The observed endocyclic angles are the same within 0·5° for the *o*-nitrobenzoic acid and the ligand. Likewise the r.m.s. deviation between observed and calculated angles is 1·2° for both the acid and the ligand. The large value arises because the calculated values are interchanged relative to the observed values, for the angles C(14)—C(15)—C(10) and C(13)—C(14)—C(15) in the ligand as well as for the respective angles in the acid. The interchange results in r.m.s. values of 0·4 and 0·7° for the ligand and the acid, respectively. Deviation is also considerable for the angle C(11)—C(12)—C(13) and for the corresponding angle in the acid, the calculated value being about 1·0° smaller than the observed value in each case.

The best agreement for the other ligand is obtained when COO^- is an orthogonal but NO_2 is a coplanar substituent (Table 3). The r.m.s. deviation between observed and calculated angles is 0·5°, and calculation yields a value 1·2° too high for the angle C(17)—C(22)—C(21). The deviations for the other angles are less than 0·6°.

Weak N···O and O···O hydrogen bonds link the complex units into a one-dimensional chain in the *a* direction (Fig. 2). Hydrogen-bonding parameters are given in Table 4. The intramolecular interactions N(1)—H(10)···O(7) and N(1)—H(11)···O(3) with angle values of 98 and 100°, N···O distances of 2·777 (6) and 2·809 (5) Å and H···O distances of 2·57 and 2·54 Å, respectively, could be interpreted as N···O contacts rather than hydrogen bonds and have therefore been omitted from figures and Table 4.

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Structure of Erbium Ditartrate Trihydrate, $Er^{4+}.2C_4H_4O_6{}^{2-}.3H_2O$

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Abstract. $M_r = 517·45$, tetragonal, $P4_12_12$, $a = 5·995$ (1), $b = 5·995$ (1), $c = 36·433$ (8) Å, $V = 1309·4$ Å^3, $Z = 4$, $D_x = 2·62$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 6·74$ mm^{-1}, $F(000) = 1000$, $T = 273$ K. Final $R = 0·045$ for 1704 observed reflexions. Erbium and hydrogen bonds involving water link the

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tartrate molecules into sheets parallel to (001); these sheets are linked together by hydrogen bonding between adjacent tartrate molecules.

Introduction. As part of an investigation into the synthesis and characterization of rare-earth-element tartrates, the crystal structure of erbium ditartrate trihydrate was determined.

Experimental. Equidimensional crystals by gel method (Hawthorne, Borys & Ferguson, 1982), ~0·10 mm diameter; Syntex $P2_1$ diffractometer, lattice parameters from 15 reflexions ($5<2\theta<25°$), confirmed from powder diffraction pattern; precession photographs, systematic absences $h\neq 2n(h00)$, $l\neq 4n(00l)$; 1894 reflexions ($+h$, $+k$, $+l$, $2\theta<60°$), two standard reflexions (023,218) every 48 reflexions (1·9% variation), $R_{\rm int} = 0{\cdot}026$ (for 36 pairs of equivalent reflexions); Lp and spherical absorption corrections; 1858 unique, 1704 observed (3σ) reflexions; structure solved by Patterson and Fourier methods; full-matrix least-squares refinement based on $|F|$, with anisotropic temperature factors for non-H atoms, converged at $R = 0{\cdot}045$, $wR = 0{\cdot}055$ ($w = 1$), $S = 4{\cdot}42$; maximum shift/error <0·01, final ΔF map ±0·8e Å^3; scattering factors and anomalous-dispersion corrections from Cromer & Mann (1968) and Cromer & Liberman (1970); structure refined using *RFINE* (L. W. Finger, personal communication), all other calculations done with XRAY 76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976).

Discussion. Atomic parameters are given in Table 1,* bond distances and angles in Table 2. The bond lengths and angles for the tartrate molecule are conformable with those reported for (+)-tartaric acid (Okaya, Stemple & Kay, 1966) and calcium tartrate tetrahydrate (Hawthorne *et al.*, 1982). Erbium is surrounded by nine O atoms between 2·29 and 2·62 Å. There was some difficulty locating the hydrogen positions in the final stages of refinement. Around the O(8) water, there is a general smear of density ~1 Å from O(8), suggesting that possibly the H atoms are disordered.

The intermolecular linkage is shown in Fig. 1. Erbium links the tartrate molecules into infinite sheets parallel to (001), and also bonds to three water molecules that form hydrogen bonds to adjacent tartrate molecules within the sheet. Projecting tartrate

Table 1. *Atomic coordinates and isotropic thermal parameters*

	x	y	z	B(Å^2)*
Er	0·32117 (7)	0·32117 (7)	0	1·13 (1)
C(1)	0·8323 (17)	0·2358 (14)	0·0337 (3)	1·4 (1)
C(2)	0·9634 (15)	0·1776 (19)	0·0686 (3)	1·3 (1)
C(3)	0·8737 (17)	−0·0378 (19)	0·0862 (4)	2·1 (2)
C(4)	0·5144 (20)	0·4207 (21)	0·1270 (4)	2·5 (2)
O(1)	0·6248 (12)	0·2204 (14)	0·0353 (2)	2·1 (1)
O(2)	0·9415 (11)	0·3086 (16)	0·0069 (2)	2·1 (1)
O(3)	0·1918 (14)	0·1641 (15)	0·0589 (2)	1·6 (1)
O(4)	0·8986 (16)	0·7832 (15)	0·0620 (3)	2·8 (2)
O(5)	0·5553 (19)	0·5719 (19)	0·1034 (3)	3·7 (2)
O(6)	0·4004 (16)	0·2542 (16)	0·1221 (3)	3·1 (2)
O(7)	0·3150 (21)	0·6148 (14)	0·0435 (2)	3·2 (2)
O(8)	0·6297 (15)	0·6297 (15)	0	4·2 (3)
H(2)C	0·93 (2)	0·27 (2)	0·090 (3)	1·0
H(3)C	0·74 (2)	0·02 (2)	0·101 (3)	1·0
H(3)O	0·26 (2)	0·19 (2)	0·069 (4)	1·0
H(4)O	0·85 (3)	0·75 (2)	0·046 (4)	1·0
H(7)*A*	0·17 (2)	0·65 (2)	0·044 (4)	1·0
H(7)*B*	0·37 (2)	0·72 (2)	0·027 (3)	1·0

* $B = \frac{4}{3}\sum_i\sum_j g_{ij}\beta_{ij}$; g_{ij} = real-space metric tensor.

Table 2. *Interatomic distances* (Å) *and angles* (°)

Er polyhedron			
Er–O(1) ×2	2·310 (7)	Er–O(7) ×2	2·369 (7)
Er–O(2) ×2	2·292 (7)	Er–O(8)	2·616 (7)
Er–O(3) ×2	2·468 (7)	Mean	2·388
Tartrate molecule			
C(1)–O(1)	1·249 (13)	C(3)–O(4)	1·396 (16)
C(1)–O(2)	1·252 (12)	C(3)–C(4)	1·519 (18)
C(1)–C(2)	1·536 (14)	C(4)–O(5)	1·274 (17)
C(2)–O(3)	1·416 (12)	C(4)–O(6)	1·223 (14)
C(2)–C(3)	1·538 (15)		
O(1)–C(1)–O(2)	126 (1)	C(2)–C(3)–O(4)	110 (1)
O(1)–C(1)–C(2)	117 (1)	C(2)–C(3)–C(4)	111 (1)
O(2)–C(1)–C(2)	117 (1)	O(4)–C(3)–C(4)	113 (1)
C(1)–C(2)–O(3)	108 (1)	C(3)–C(4)–O(5)	113 (1)
C(1)–C(2)–C(3)	111 (1)	C(3)–C(4)–O(6)	121 (1)
O(3)–C(2)–C(3)	113 (1)	O(5)–C(4)–O(6)	126 (1)

Hydrogen bonds

A–H···*B*	*A*–H	H···*B*	*A*···*B*	*A*–H···*B*
O(7)–H(7)*A*···O(4)	0·9 (1)	1·9 (1)	2·78 (2)	155 (12)
O(7)–H(7)*B*···O(2)	0·9 (1)	1·9 (1)	2·69 (1)	145 (12)
O(7)–H(7)*B*···O(8)	0·9 (1)	1·9 (1)	2·47 (1)	117 (11)

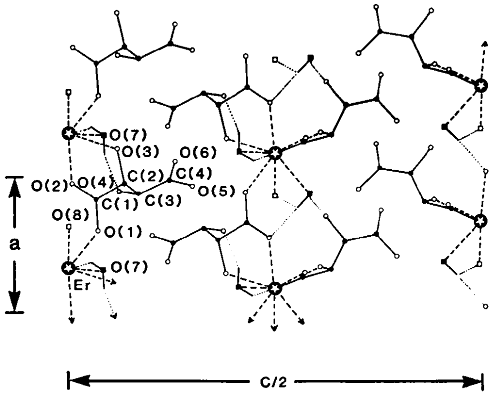

Fig. 1. A projection of the erbium ditartrate trihydrate structure down the y axis; intersheet hydrogen bonds have been omitted for clarity. H(7)*B* hydrogen bonds to O(8) that is occluded by Er. Er is represented by white stars, C by small filled circles, O by small hollow circles, and water molecules [O(7) and O(8)] by small squares.

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* Lists of structure factors, anisotropic thermal parameters and the magnitudes and orientations of the axes of the thermal ellipsoids have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38318 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

molecules from neighbouring sheets mesh together (Fig. 1) and form hydrogen bonds to each other to provide the intersheet linkages.

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The Structure of Pentacarbonyl(5,6-dihydro-4*H*-1,2-benzodithiole-7-carbothialdehyde-*S*^{7})tungsten(0), [W($C_8H_8S_3$)(CO)$_5$]

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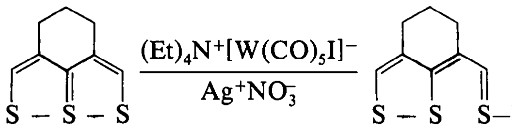
Abstract. $M_r = 524\cdot24$, triclinic, $P\bar{1}$, $a = 8\cdot337$ (2), $b = 9\cdot039$ (3), $c = 10\cdot962$ (3) Å, $\alpha = 90\cdot83$ (3), $\beta = 91\cdot21$ (3), $\gamma = 100\cdot05$ (3)°, $U = 813\cdot08$ Å^3, $D_x = 2\cdot141$ Mg m^{-3} for $Z = 2$, $F(000) = 496$, Mo $K\alpha$ radiation, $\lambda = 0\cdot71069$ Å, μ(Mo $K\alpha$) = 7·828 mm^{-1}, $R = 0\cdot0454$ for 3503 reflections with $F_o \geq 6\sigma(F_o)$. The S–S and S···S distances of 2·078 (4) and 2·752 (3) Å show that, in the solid state, the compound has a bicyclic structure, not a tricyclic one.

Introduction. Treatment of (I) with $(Et)_4N^+[W(CO)_5I]^-$ in CH_2Cl_2 solution in the presence of aqueous $AgNO_3$ gave the title compound(II) (Pogorzelec & Reid, 1983). Crystals suitable for X-ray investigation were grown from benzene/hexane solution.



(I) (II)

Experimental. Stoe STADI-2 two-circle diffractometer, graphite-crystal-monochromatized Mo $K\alpha$ radiation, crystal dimensions 0·462 × 0·103 × 0·250 mm, bounded by the ±(100), ±(010) and ±(001) faces, and mounted about **a**; 3943 reflections collected in the hemisphere $+h, \pm k, \pm l$ with $2\cdot5 \leq \theta \leq 30°$, $h = 0$–11 ($0 \leq \mu \leq 27\cdot960°$), ω–2θ scan mode, step width of 0·01° in ω, a time of 1·0s per step, 160 steps per scan; backgrounds measured at both ends of the scan for 80·0s each; standard reflections measured every 50 reflections showed only small random deviations from their means; absorption corrections were applied.

The structure was solved and refined using *SHELX* 76 (Sheldrick, 1976). The W atom position was found on a Patterson synthesis and a subsequent difference synthesis gave the positions of all the other non-H atoms. Least-squares refinement with the matrix blocked so that the W atom refined every cycle, but the CO groups and $C_8H_8S_3$ group refined in alternate cycles (for computational economy), with anisotropic temperature parameters for all non-H atoms and the H atoms in calculated positions ($d_{\text{C-H}} = 1\cdot08$ Å) with a common isotropic temperature parameter, converged to give an R of 0·0454 and $R_G [= (\sum w\Delta^2/\sum wF_o^2)^{1/2}]$ of 0·0566 for 3503 independent reflections with $F_o \geq 6\sigma(F_o)$. In the final cycles 200 parameters were varied, comprising 66 positional coordinates, 132 anisotropic temperature factor components, one common isotropic temperature factor and one overall scale factor: a final difference map showed no significant residual features. Complex neutral-atom scattering factors were used for all atoms (Cromer & Waber, 1974). The reductions in R_G at all stages of the refinement were significant at the 99·5% level (Hamilton, 1965).†

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† Lists of structure factors, anisotropic thermal parameters and calculated H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38328 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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