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Structure of Monoclinic Sodium (Ethylenediaminetetraacetato)holmate(III) Octahydrate, Na[Ho(C₁₀H₁₂N₂O₈)]·8H₂O: Disorder Induced by Radiation

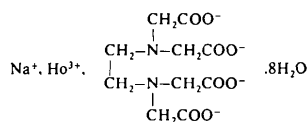
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Abstract. $M_r = 620.3$, monoclinic, $Fd\bar{1}1$, $a = 19.333(3)$, $b = 35.366(6)$, $c = 12.106(2)$ Å, $\alpha = 90.40(10)^\circ$, $V = 8277.0$ Å³, $Z = 16$ [conventional setting: Cc , $a = 12.106(2)$, $b = 19.333(3)$, $c = 18.650(3)$ Å, $\beta = 108.54(10)^\circ$, $Z = 8$], $D_x = 1.991$ g cm⁻³, $Mo K\alpha$, $\lambda(\alpha_1) = 0.70930$ Å, $\mu = 39.7$ cm⁻¹, $F(000) = 4924$, $T = 294$ K, $R = 0.026$ for 4843 reflections. The atomic positions are very similar to those in the analogous compounds of lighter lanthanoid elements which crystallize in the orthorhombic space group $Fdd2$. The monoclinic symmetry comes from ordering of water molecules in alternate sites which are occupied randomly in the orthorhombic structure. Exposure to X-rays induces disorder which makes the crystals more nearly orthorhombic. The Dy compound of the same composition is isomorphous with $a = 19.358(3)$, $b = 35.408(3)$, $c = 12.103(5)$ Å, $\alpha = 90.34(3)^\circ$.

Introduction. Crystal structure parameters were desired for the title salt,



so that it could be used to measure anomalous-scattering terms for Ho at various wavelengths

(Chapuis, Templeton & Templeton, 1985) by analysis of diffraction intensities. We had expected the crystals to have the orthorhombic structure with space group $Fdd2$ which has been reported for the analogous salts of Pr, Nd, Sm, Gd, Tb, Dy and Er (Hoard, Lee & Lind, 1965; Koetzle & Hamilton, 1975; Nassimbeni, Wright, van Niekerk & McCallum, 1979; Templeton, Templeton, Zalkin & Ruben, 1982). Indeed, the diffraction patterns are very similar to those of the orthorhombic crystals, but close inspection reveals monoclinic symmetry with twice as large an asymmetric unit. We describe the structure in space group $Fd\bar{1}1$ (an unconventional setting of Cc) because that gives cell dimensions and atomic coordinates which are nearly the same as for $Fdd2$. We find the same monoclinic structure for crystals of the Dy compound, contrary to the findings of Nassimbeni *et al.* (1979). A recent neutron-diffraction study by Engel, Takusagawa & Koetzle (1984) of the orthorhombic Sm compound found disorder of water molecules among two sets of sites. The monoclinic symmetry involves an ordering of the occupancy of these same sites.

Experimental. The compound was made from Ho₂O₃ (Research Chemical Corporation) dissolved in HCl and mixed with a hot solution of Na₂H₂(edta)·2H₂O (Aldrich Chemical Company); pH adjusted to about 5 with NaOH; slow evaporation, then recrystallization

from water yielded crystals, many of which were twinned in a manner nearly to superimpose the reflections which are equivalent in orthorhombic symmetry (Dy compound made in the same way). Cell dimensions from 24 reflections in θ range 57 to 60° measured with Cu $K\alpha$ and CAD-4 diffractometer. Another crystal 0.15 × 0.24 × 0.25 mm, 15 faces. Picker FACS-I diffractometer. Cell dimensions from 12 reflections in θ range 20 to 22°. Analytical absorption correction 1.42 < A < 1.71. Max. $(\sin\theta)/\lambda = 0.60 \text{ \AA}^{-1}$. hkl range 0 to 12, -42 to 42, -14 to 14. 10% intensity-decay correction based on average of the changes of 400 (14%), 555 (2%), and 044 (13%). 4977 unique reflections; 91 with $I < \sigma$ and 43 others with $(\sin\theta)/\lambda < 0.11 \text{ \AA}^{-1}$ given zero weight. Refinement started with known structure of Sm salt duplicated for monoclinic symmetry, refined on F . 24 H atoms included in refinement with each thermal parameter equal to that of adjacent C atom and with distance restraints C-H = 1.00 (5), H-H = 1.63 (10), C'-H = 2.06 (10), N'-H = 2.04 (10) Å (C' and N' next atoms in chain); water H atoms omitted. 330 parameters including f'' for Ho and anisotropic thermal parameters for Ho and water O atoms, but not counting those of H atoms. $R = 0.026$, $wR = 0.034$, $S = 1.1$, $w = [\sigma(F)]^{-2}$, $p = 0.05$ in calculation of $\sigma(F^2)$. Max. $\Delta/\sigma = 0.16$. $|\Delta\rho|_{\max} = 0.7 \text{ e \AA}^{-3}$. Max. correction for extinction 6% of F . Atomic f for Ho³⁺, Na⁺, O^{0.5-} (interpolated), and neutral O, N, C, and H from *International Tables for X-ray Crystallography* (1974). Local unpublished programs and ORTEP (Johnson, 1965). Atomic parameters are listed in Table 1. For this setting the equivalent positions are: $x, y, z; \frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} + z$; plus the translations of the F lattice. The origin of this doubly polar space group was defined by placing the center of gravity of the two Ho atoms at $(x, \frac{1}{2}, 0)$ in order to reduce covariance terms and to improve convergence (Templeton, 1960). The correct absolute polarity is confirmed by the refinement of f'' for Ho to the positive value 4.92 (10) e/atom. Cell dimensions for Dy compound from 13 reflections, CAD-4, Mo $K\alpha$.*

Discussion. The structure consists of two formula units at positions which are related approximately by a transformation $(\frac{1}{2} - x, \frac{1}{2} - y, z)$ which corresponds to a twofold axis in the c direction. Deletion of this symmetry element from $Fdd2$ lowers the space group to $Fd11$. For most of the atoms this relation is accurate to better than 0.2 Å, and the structure is hardly different from that found for the orthorhombic compounds of the lighter lanthanoid elements. The only major difference

Table 1. Atomic parameters

$$B_{eq} = \frac{1}{3} \sum_i \sum_j B_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	B or $B_{eq} (\text{\AA}^2)$
Ho(1)	0.08268 (2)	0.15544 (1)	-0.00108 (1)	1.55 (1)†
Ho(2)	0.41843 (2)	0.34456	0.00108	1.57 (1)†
H(1)	0.1345 (3)	0.1100 (2)	-0.1220 (4)	2.3 (2)†
H(2)	0.1308 (3)	0.10709 (14)	0.1208 (4)	2.1 (2)†
H(3)	0.0014 (4)	0.09946 (13)	-0.0121 (4)	2.6 (2)†
H(4)	0.0289 (7)	0.1245 (4)	0.3801 (14)	14.1 (7)†
H(5)	0.0615 (4)	0.0409 (2)	0.1002 (5)	3.9 (2)†
H(6)	0.1438 (6)	0.0988 (2)	0.5612 (9)	7.5 (3)†
H(7)	0.1354 (9)	0.1830 (3)	0.4503 (7)	11.6 (5)†
H(8)	0.0072 (6)	0.0509 (3)	0.2942 (8)	9.7 (4)†
H(9)	0.3656 (4)	0.38783 (14)	-0.1232 (4)	2.6 (2)†
H(10)	0.3694 (4)	0.39352 (14)	0.1195 (4)	2.4 (2)†
H(11)	0.4962 (4)	0.40195 (13)	-0.0141 (4)	2.5 (2)†
H(12)	0.4824 (5)	0.3903 (3)	0.3226 (9)	7.7 (4)†
H(13)	0.4374 (4)	0.4615 (2)	0.1033 (5)	3.7 (2)†
H(14)	0.3476 (6)	0.4025 (2)	0.5656 (9)	7.8 (4)†
H(15)	0.3851 (5)	0.3232 (2)	0.4567 (5)	6.0 (3)†
H(16)	0.4832 (6)	0.4746 (3)	0.3226 (9)	8.5 (4)†
Na(1)	0.1393 (2)	0.13492 (9)	0.3115 (3)	3.84 (7)
N(1)	0.0553 (5)	0.2273 (2)	0.0058 (5)	2.0 (1)
N(2)	0.1482 (4)	0.1922 (2)	-0.1603 (5)	1.9 (1)
O(1)	0.0276 (3)	0.16832 (14)	0.0724 (4)	2.51 (9)
O(2)	0.0967 (3)	0.18267 (13)	0.1789 (4)	2.12 (9)
O(3)	0.2013 (3)	0.16792 (13)	0.0299 (4)	2.30 (9)
O(4)	0.0174 (3)	0.16333 (13)	-0.1622 (4)	1.95 (9)
O(5)	-0.1011 (4)	0.2088 (2)	0.1479 (6)	4.5 (1)
O(6)	0.1072 (4)	0.2319 (2)	0.2897 (5)	3.3 (1)
O(7)	0.3100 (5)	0.1760 (2)	-0.0271 (5)	3.7 (1)
O(8)	0.0059 (4)	0.1708 (2)	-0.3444 (5)	3.0 (1)
C(1)	0.0756 (5)	0.2464 (2)	-0.1000 (7)	2.4 (2)
C(2)	0.1462 (5)	0.2337 (2)	-0.1359 (6)	2.3 (1)
C(3)	0.0181 (6)	0.2327 (2)	0.0212 (7)	3.2 (2)
C(4)	0.0504 (5)	0.2007 (2)	0.0866 (6)	2.6 (1)
C(5)	0.0951 (6)	0.2441 (2)	0.1007 (7)	2.6 (2)
C(6)	0.0992 (5)	0.2169 (2)	0.1979 (6)	2.0 (1)
C(7)	0.2220 (6)	0.1798 (2)	-0.1623 (6)	2.5 (1)
C(8)	0.2470 (6)	0.1743 (2)	-0.0446 (6)	2.3 (1)
C(9)	0.1142 (5)	0.1832 (2)	-0.2663 (7)	2.7 (2)
C(10)	0.0406 (5)	0.1723 (2)	-0.2570 (6)	2.1 (1)
Na(2)	0.3656 (2)	0.36895 (9)	0.3133 (3)	3.60 (7)
N(3)	0.4472 (5)	0.2724 (2)	0.0095 (5)	2.0 (1)
N(4)	0.3529 (4)	0.3056 (2)	-0.1567 (4)	1.8 (1)
O(9)	0.5293 (3)	0.33353 (13)	0.0733 (4)	2.53 (9)
O(10)	0.4052 (3)	0.31877 (13)	0.1810 (4)	2.11 (9)
O(11)	0.2991 (3)	0.33121 (13)	0.0332 (4)	2.42 (9)
O(12)	0.4829 (3)	0.33606 (13)	-0.1604 (4)	1.99 (9)
O(13)	0.6037 (5)	0.2941 (2)	0.1490 (7)	5.3 (2)
O(14)	0.3965 (3)	0.2701 (2)	0.2957 (4)	2.9 (1)
O(15)	0.1916 (5)	0.3210 (2)	-0.0227 (5)	3.5 (1)
O(16)	0.4931 (4)	0.3282 (2)	-0.3434 (4)	3.0 (1)
C(11)	0.4271 (5)	0.2523 (2)	-0.0945 (6)	2.1 (1)
C(12)	0.3557 (5)	0.2645 (2)	-0.1301 (6)	2.2 (1)
C(13)	0.5218 (6)	0.2689 (2)	0.0244 (7)	3.1 (2)
C(14)	0.5534 (6)	0.3009 (2)	0.0879 (6)	2.7 (1)
C(15)	0.4100 (6)	0.2560 (2)	0.1055 (7)	2.5 (2)
C(16)	0.4031 (5)	0.2844 (2)	0.2017 (6)	2.0 (1)
C(17)	0.2798 (6)	0.3173 (2)	-0.1562 (6)	2.5 (1)
C(18)	0.2543 (6)	0.3232 (2)	-0.0399 (6)	2.2 (1)
C(19)	0.3869 (5)	0.3131 (2)	-0.2625 (6)	2.6 (1)
C(20)	0.4606 (5)	0.3264 (2)	-0.2550 (5)	1.6 (1)

is the locations and hydrogen bonding of a few of the water molecules. The above transformation fails by 0.46 Å for the $W(7)$ – $W(15)$ pair, 0.91 Å for $W(4)$ – $W(12)$ and 1.07 Å for $W(8)$ – $W(16)$. The last two pairs correspond to those which are distributed randomly in alternate sites in the Sm structure, and designated as 4B, 4A and 8B, 8A, respectively, by Engel, Takusagawa & Koetzle (1984) in their neutron-diffraction study. In that work 18 hydrogen bonds were identified. Taking account of the half-occupancy of some sites, these become 28 distinct bonds in the monoclinic symmetry. We find exactly the same bonds, on the basis of O-atom positions, plus one more: $W(4)$ – $W(14) = 2.75 (2) \text{ \AA}$.

* Tables of structure factors, anisotropic thermal parameters, H-atom coordinates, and bond distances have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39877 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

In the Sm salt the corresponding distance was found to be 3.04 Å, but an H atom [designated as 'U(4B)'] is pointed in about the right direction for such a bond. The network of bonds near the *c* axis, where the higher symmetry breaks down, is shown in Fig. 1. Water molecules *W*(8) and *W*(16), which are equivalent in *Fdd2*, form hydrogen bonds to *W*(15) and O(5), atoms which are not equivalent even in chemical state. This unsymmetrical bonding pulls them and their neighbors away from locations which conform to the higher symmetry. The only other lack of *Fdd2* symmetry in the topology of the bonds is in the chain *W*(12)–H...*W*(16)–H...*W*(8)–H...*W*(4), if the H atoms are assigned according to the neutron-diffraction study; an extra H atom on *W*(4) is not involved in a bond.

Hydrogen-bond O–O distances (deposited) range from 2.64 to 2.85 Å, except *W*(8)–*W*(4) = 2.88 (2) and *W*(12)–*W*(16) = 2.98 (1) Å. These two bonds join the atoms which are most involved in the order-disorder transition discussed below, and these distances may be affected by it.

Exposure to X-rays causes the unit-cell angle α to become more nearly equal to 90°, with insignificant

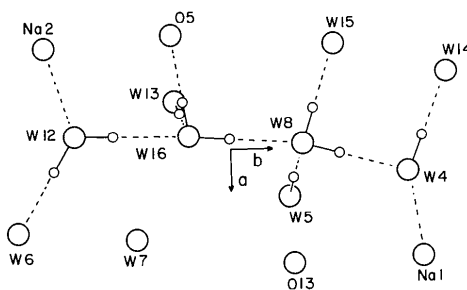


Fig. 1. Hydrogen bonding near the *c* axis. H atoms are shown at calculated positions. In the model for the radiation-induced disorder, some of the bonds to O(5) and *W*(15) are broken, new bonds are formed to *W*(7) and O(13), and the average structure approaches orthorhombic symmetry with a twofold axis at the center of the figure.

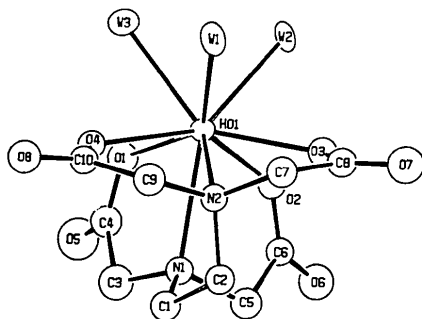


Fig. 2. Coordination of Ho(1) and atomic labeling of its neighbors. Labels for the corresponding neighbors of Ho(2) are obtained by adding 2 for N, 8 for O or water, and 10 for C.

Table 2. Neighbor distances (Å) for holmium

Ho(1)	O(1)	2.353 (6)	Ho(2)	O(9)	2.348 (6)
	O(2)	2.391 (5)		O(10)	2.381 (5)
	O(3)	2.365 (7)		O(11)	2.387 (7)
	O(4)	2.341 (5)		O(12)	2.335 (5)
	W(1)	2.387 (5)		W(9)	2.383 (5)
	W(2)	2.449 (5)		W(10)	2.432 (5)
	W(3)	2.531 (6)		W(11)	2.533 (6)
	N(1)	2.597 (6)		N(3)	2.613 (6)
	N(2)	2.655 (6)		N(4)	2.667 (6)

changes in axial lengths. As noted above, the diffraction intensities tend to decrease, but not uniformly. At the end of the data collection α was 90.28°. For another crystal, after much exposure to X-rays, α was 90.11°. Values of 90.49 (1) and 90.45 (2)° were measured for two other fresh crystals. We attribute these changes to the introduction of disorder in the hydrogen bonding, with an approach to the symmetry of the *Fdd2* structure. Since our specimen suffered some damage in the course of the experiment, the results include the effects of some of this disorder, for example in the highly anisotropic thermal parameters for some of the water molecules.

Cell dimensions of the Dy compound (*Abstract*) show that it is also monoclinic. Numerous cell determinations of crystals of the Pr, Sm and Gd compounds gave angles very close to 90° and no indication of monoclinic symmetry. It is possible that different methods of crystallization would produce ordered specimens of these materials, or that the monoclinic structure occurs in salts which have not been studied carefully, particularly those of Tb and Er.

Each Ho³⁺ ion is complexed by six atoms from a single edta group and also three water molecules, as depicted for Ho(1) in Fig. 2. A similar drawing for Ho(2) is almost indistinguishable at this scale. Distances from each Ho atom to its nine neighbors, listed in Table 2, are similar to those reported for the orthorhombic crystals (Templeton, Templeton, Zalkin & Ruben, 1982), but slightly smaller because of the lanthanoid contraction of ionic radius; on the average they are 0.027 Å less than in the Gd salt. Bond distances within the edta moiety (deposited) are normal; the average values for the various chemical types of bond [N–C = 1.482 (5), C–O = 1.252 (4), C–C = 1.505 (5) (ethylenediamine) and 1.510 (7) Å (acetate)] agree within 0.006 Å with averages from the Pr, Sm and Gd structures (Templeton *et al.*, 1982).

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Structure of μ -Oxo-bis[(2-hydroxyethanesulfonato)triphenylantimony(V)], [Sb₂O(C₂H₅O₄S)₂(C₆H₅)₆]

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Abstract. $M_r = 972.39$, monoclinic, $P2_1/c$, $a = 10.861$ (4), $b = 19.916$ (6), $c = 9.075$ (4) Å, $\beta = 94.15$ (5)°, $U = 1958$ Å³, $Z = 2$, $D_x = 1.649$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.56$ mm⁻¹, $F(000) = 972$, $T = 294$ (1) K. Final $R = 0.032$ for 2727 unique observed X-ray diffractometer data. Two only slightly distorted trigonal-bipyramidal units (μ -O)—SbPh₃—O—S(O₂)C₂H₄OH with O atoms in apical and C(phenyl) atoms in equatorial positions are linked *via* a linear Sb—O—Sb bridge. The Sb— μ -O bond of 1.936 (1) Å is rather short. The Sb—O bond to the unidentately coordinated sulfonate group [2.276 (4) Å] appears to have appreciably high ionic character.

Introduction. The chemistry of triorgano-Group V element sulfonates is rather unexplored and structures of such compounds are unknown. We recently obtained for the first time, by reaction of Ph₃SbO (Ph = C₆H₅) with sulfonic acids in different solvents, antimony compounds which correspond to the formula (Ph₃SbO₃SR)₂O (HO₃SR = sulfonic acid) having, according to spectroscopic data, an Sb—O—Sb bridge. Compounds of the type (R₃SbX)₂O (R = organogroup, X = anionic ligand) may have a bent Sb—O—Sb bridge, as in (Ph₃SbN₃)₂O (Sb—O—Sb:139.8°, Ferguson & Ridley, 1973), or a linear bridge as in (Ph₃SbOOC₄H₉)₂O (Starikova, Shchegoleva, Trunov & Pokrovskaya, 1978). To obtain more facts about influences which determine the type of bridge in distiboxane and also to learn more about the coor-

dination of the sulfonate group with organoelement moieties (a field which has also been neglected) we started to determine the crystal and molecular structure of organoantimony sulfonates and report here the structure of (Ph₃SbO₃SCH₂CH₂OH)₂O.

Experimental. (Ph₃SbO₃SCH₂CH₂OH)₂O crystallizes from a solution of 10 mmol isethionic acid and 10 mmol Ph₃SbO in 250 ml acetone after standing for 1 d. Crystal size 0.32 × 0.29 × 0.10 mm, $\omega/2\theta$ scan, scan speed 0.04–0.01° s⁻¹ in θ , Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from least-squares fit with 25 reflexions in range $9.3 \leq \theta \leq 14.0^\circ$; three standard reflexions recorded every 4 h, only random deviations; 3799 reflexions, $1 \leq \theta \leq 25^\circ$, $-10 \leq h \leq 10$, $0 \leq k \leq 23$, $0 \leq l \leq 12$; after averaging 3541 unique reflexions, 2727 with $I > 3\sigma(I)$, Lorentz-polarization correction and absorption correction *via* ψ scans; systematic absences: $(h0l) l = 2n+1$, $(0k0) k = 2n+1$, space group $P2_1/c$; structure solution *via* Patterson function, ΔF syntheses and full-matrix least-squares refinements with anisotropic temperature factors for all non-H atoms; complex neutral-atom scattering factors from Cromer (1974) and Cromer & Waber (1974). H atoms placed in geometrically calculated positions (C—H 1.08 Å) and not included in structure-factor calculation. Least-squares refinement based on F and 241 refined parameters, $R = 0.032$, unit weights; max. Δ/σ for all parameters 0.02, av. < 0.00; no significant features in final ΔF synthesis; programs: Enraf-Nonius SDP (1978), ORTEP (Johnson, 1976) and POP1 (van de Waal, 1976).

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