Crystal Growth and Structural Study of the New Series Ln(OH)CrO$_4$ (Ln = Y, Dy–Lu): Crystal Structure of Er(OH)CrO$_4$

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Single crystals of the new series Ln(OH)CrO$_4$ (Ln = Y, Dy–Lu) have been obtained by hydrothermal procedures. The structure of Er(OH)CrO$_4$ has been determined by single-crystal X-ray techniques. The compound has monoclinic symmetry, space group $P2_1/n$, $Z = 8$, with $a = 8.106(3)$, $b = 11.324(2)$, $c = 8.251(1)$ Å, $\beta = 94.14(2)°$ and $V = 755.4(3)$ Å^3. Final R values were $R = 0.034$, $R_w = 0.049$, for 2207 observed reflections. X-ray powder data show that all compounds of the title series are isomorphous. The coordination polyhedron of the lanthanide cations can be considered a square antiprism, with hydrogen bonds linking CrO$_4$ and LnO$_8$ groups. The X-ray data in this series provide evidence for the lanthanide contraction. © 1991 Academic Press, Inc.

Introduction

Within the studies of the system Ln–O–X, two kinds of compounds with different stoichiometries are known. One type corresponds to the formula Ln(OH)$_2X$, in which X is a monovalent anion (F, Cl, Br, or NO$_3$) and the other type corresponds to the formula Ln(OH)X where X is a divalent anion (CO$_3$, SO$_4$, or CrO$_4$).

For the compounds Ln(OH)$_2X$ only one structural type has been described, for all the X anions and for the lanthanides from La to Gd (*1–12*). However, this system has not been investigated for the smaller lanthanide cations.

In the Ln(OH)X series with $X = $ CO$_3$, the structure has been described for $Ln = $ Pr–Nd, where both compounds are isomorphic with hexagonal symmetry, and for $X = $ SO$_4$, $Ln = $ La and Pr have been studied; only one monoclinic structural type has been reported for these larger lanthanides (*13, 14*).

In our previous studies on the Ln(OH)CrO$_4$ system, we encountered an isomorphous series for $Ln = $ La–Nd; we determined the structure (*15*) and studied the magnetic and spectroscopic properties (*16*). In addition we found a second isomorphous series for the lanthanides cations of intermediate size, $Ln = $ Eu–Tb (*17*). In the

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present work we have found a third isomorphous series for the lanthanides with smaller radii, Ln = Dy–Lu.

Experimental

Crystal Growth

Single crystals of the phases $Ln(OH)CrO_4$ (Ln = Y, Dy, Er, Yb, Lu) have been grown by a hydrothermal procedure using closed glass tubes heated isothermally at 150°C for 1 week.

The dimensions of the tubes were 20 cm in height and 5 cm in diameter. The tubes were filled up to half of their total volume; after heating, they were cooled slowly.

As reagents we have used the corresponding Ln_2O_3 and $K_2Cr_2O_7$ in the molar ratio 1 : 10. Under these conditions, needle-shaped and red-orange-colored single crystals of all the phases were formed on the bottom of the tubes.

Crystal Structure Data

Er(OH)CrO$_4$: M = 300.26, monoclinic, a = 8.106(3), b = 11.324(2), c = 8.251(1) Å, β = 94.14(2)°, U = 755.4(3) Å^3, Z = 8, D = 5.28 g · cm^{-3}, $F(000)$ = 1064, (MoKα) = 250.8 cm^{-1}, space group $P2_1/n$, T = 295 K, R = 0.034, R_w = 0.049 for 2207 observed reflections.

Data Collection

A prismatic crystal of Er(OH)CrO$_4$ was mounted on an Enraf Nonius CAD4-F automatic diffractometer. The cell dimensions were refined by least-squares fitting of the setting angles of 25 reflections.

The intensities of all 2555 reflections with $1 < \theta < 30$ and the range from $(-11, 0, 0)$ to $(11, 15, 11)$ were measured at 295 K using monochromatic MoKα radiation (λ = 0.71069 Å) and the $\omega/2\theta$ scan technique.

Three reflections monitored periodically during data collection revealed no crystal decomposition. The intensities were corrected for Lorentz and polarization effects;

2207 reflections were considered as observed with $I > 26(I)$. Scattering factors for neutral atoms and anomalous dispersion corrections for Er and Cr were taken from the International Tables for X-Ray Crystallography (18).[1]

The X-Ray powder patterns were recorded using a Siemens Kristalloflex 810 diffractometer and a D-500 goniometer with nickel-filtered copper radiation (λ = 1.540598 Å) selected with a graphite monochromator. The d spacings measurements were made at a scanning rate of 0.1° · 2θ cm^{-1}, using silicon (a = 5.430881 Å) as an internal standard.

Structure Determination

The structure was solved by Patterson and Fourier methods. An empirical absorption correction was applied at the conclusion of the isotropic refinement. The H atoms were located in a final difference Fourier synthesis as the highest peak of the map and their coordinates were subsequently refined. In order to prevent bias on F vs F_0 or sin θ/λ, the last steps of the refinement were carried out with weights w = $1/(a + b|F_0|)^2$, where a = 3.93, b = 0.04 if $|F_0| > 50$ and a = 0.67, b = 0.02 if $|F_0| < 50$, calculated by PESOS (20). Final refinement with fixed isotropic temperature factors for H atoms gave R = 0.034 and R_w = 0.049. The average shift/error is 0.059. Most of the calculations were carried out with the X-Ray 80 (21).

Results and Discussion

X-ray powder data show that the structure of this new series prevails for yttrium and for the lanthanides from dysprosium to lutetium.

Table I shows the calculated crystallographic data in which the decrease of the

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[1] Listings of structure factors and anisotropic thermal parameters have been deposited with the editor.

TABLE I

CRYSTAL DATA FOR $Ln(OH)CrO_4$ WITH ESTIMATED STANDARD DEVIATIONS IN PARENTHESES

Lanthanide	a (Å)	b (Å)	c (Å)	β (°)	V (Å^3)	D_x (g · cm^{-3})
Y	8.113(5)	11.340(3)	8.260(4)	93.97(2)	758.2	3.88
Dy	8.149(2)	11.367(1)	8.295(3)	93.91(1)	766.7(1)	5.11
Er[a]	8.106(3)	11.324(2)	8.251(1)	94.14(2)	755.4(3)	5.28
Yb	8.069(1)	11.310(1)	8.190(4)	94.25(3)	745.5(2)	5.45
Lu	8.061(2)	11.278(2)	8.165(2)	94.33(2)	740.2(3)	5.52

[a] Single crystal parameters.

parameters can be observed from Dy to Lu due to the lanthanide contraction. Table II provides d-spacings and relative intensities of the new compounds.

The structure has been solved for $Er(OH)CrO_4$. Table III lists the final parameters and Table IV provides bond lengths and angles.

Each erbium atom is bonded to eight oxygen atoms (Fig. 1) (22). The coordination polyhedron can be considered as a distorted quadrangular antiprism in which five
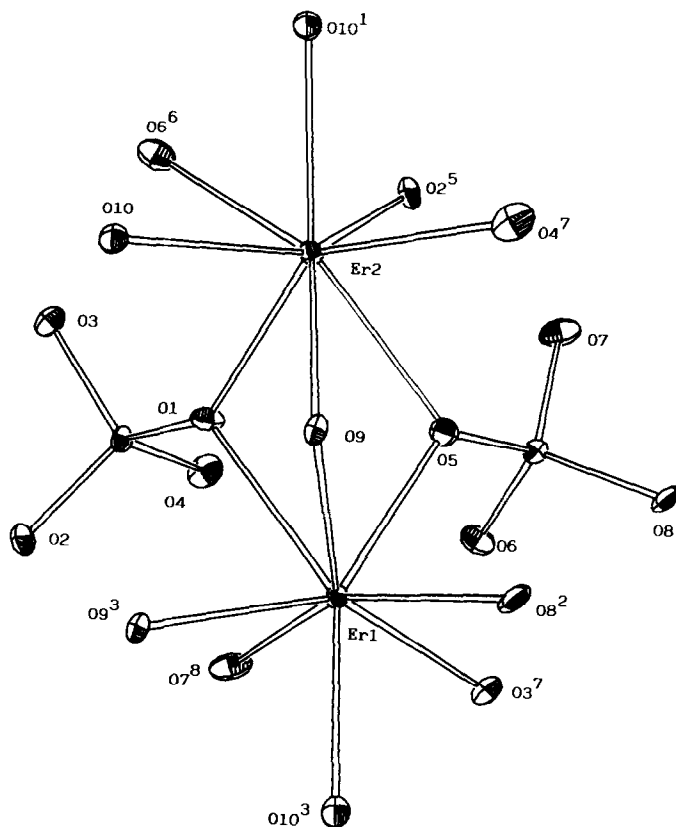

FIG. 1. Coordination polyhedron of the Er atoms.

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TABLE II
Interplanar Spacings of $Ln(OH)CrO_4$ (Ln = Y, Dy–Lu)

h k l	Y d_0	Y d_c	Y I_0	Dy d_0	Dy d_c	Dy I_0	Er d_0	Er d_c	Er I_0	Yb d_0	Yb d_c	Yb I_0	Lu d_0	Lu d_c	Lu I_0
0 1 1	6.667	6.667	40	6.692	6.691	31	6.642	6.657	42	6.602	6.521	35	6.597	6.602	31
0 2 0	5.684	5.670	16	5.691	5.684	11	5.655	5.662	14	5.655	5.655	14	5.666	5.639	12
0 2 1	4.666	4.671	65	4.683	4.695	55	4.663	4.664	74	4.647	4.649	67	4.644	4.636	58
0 1 2	3.867	3.872	52	3.892	3.888	46	3.863	3.867	46	3.842	3.841	46	3.835	3.829	43
$\bar{2}$ 1 1	3.548	3.549	25	3.555	3.563	18	3.548	3.548	24	3.534	3.534	17	3.522	3.530	17
0 3 1	3.436	3.436	46	3.450	3.445	39	3.432	3.431	46	3.421	3.423	38	3.410	3.413	35
2 1 1	3.373	3.376	35	3.385	3.391	25	3.369	3.369	35	3.354	3.351	26	3.350	3.343	26
0 2 2	3.326	3.333	82	3.325	3.345	89	3.328	3.328	73	3.314	3.311	69	3.297	3.301	65
$\bar{2}$ 2 1	3.121	3.120	28	3.123	3.121	26	3.121	3.119	32	3.110	3.108	30	3.009	3.103	21
2 2 1	3.006	3.001	30	3.015	3.013	13	2.995	2.995	24	2.983	2.981	14	2.973	2.974	15
$\bar{2}$ 1 2	2.894	2.894	10	2.908	2.905	8	2.895	2.894	13	2.885	2.880	9	2.874	2.876	8
0 3 2	2.786	2.786	45	2.791	2.794	39	2.782	2.781	46	2.769	2.770	40	2.762	2.762	43
0 1 3	2.672	2.670	100	2.683	2.681	100	2.666	2.666	100	2.648	2.647	100	2.639	2.639	100
1 3 2	2.600	2.599	9	2.606	2.606	6	2.588	2.591	6	2.574	2.579	6	2.571	2.572	5
2 2 2	2.506	2.507	7	2.512	2.512	5	2.503	2.499	6	2.485	2.485	5	2.472	2.477	7
$\bar{1}$ 2 3	2.413	2.410	6	2.418	2.416	5	2.407	2.405	6	2.397	2.392	5	2.389	2.385	5
2 3 2	2.250	2.248	7	2.257	2.255	6	2.245	2.241	7	2.228	2.230	6	2.222	2.224	6
0 3 3	2.225	2.224	11	2.231	2.230	8	2.221	2.219	6	2.208	2.207	6	2.203	2.201	8
1 4 2		2.222			2.228			2.217			2.209			2.202	
0 5 1	2.189	2.188	8	2.189	2.192	10	2.188	2.184	7	2.179	2.180	8	2.174	2.174	7
$\bar{1}$ 3 3	2.176	2.177	12	2.180	2.182	11	2.171	2.172	11	2.163	2.162	9	2.158	2.156	9
$\bar{2}$ 2 3		2.174			2.179			2.170			2.160			2.155	
0 0 4	2.063	2.063	13	2.066	2.069	10	2.056	2.057	10	2.045	2.042	11	2.040	2.036	9
0 1 4	2.028	2.030	9	2.038	2.036	6	2.022	2.024	9	2.012	2.009	8	2.005	2.003	6
$\bar{3}$ 3 2	1.990	1.988	22	1.992	1.992	21	1.981	1.984	23	1.976	1.977	24	1.974	1.974	20
0 5 2		1.988			1.993			1.984			1.979			1.973	
$\bar{1}$ 4 3	1.942	1.941	20	1.947	1.945	19	1.936	1.937	15	1.993	1.929	16	1.926	1.924	15
$\bar{2}$ 5 1		1.940			1.944			1.937			1.933			1.928	
4 2 0	1.909	1.908	7	1.915	1.914	6	1.904	1.903	7	1.895	1.895	4	1.893	1.893	4
4 1 1		1.909			1.916			1.903			1.894			1.891	
1 4 3	1.897	1.897	10	1.904	1.902	9	1.891	1.891	11	1.882	1.883	11	1.877	1.877	9
3 3 2		1.896			1.902			1.890			1.880			1.876	
$\bar{2}$ 1 4	1.869	1.867	9	1.876	1.872	7	1.860	1.863	10	1.855	1.852	7	1.850	1.848	7
4 1 2	1.843	1.846	8	1.848	1.851	6	1.840	1.843	5	1.835	1.836	8	1.833	1.834	5
0 6 1		1.843			1.847			1.840			1.837			1.832	
1 6 0		1.841			1.845			1.838			1.835			1.830	
$\bar{2}$ 4 3	1.813	1.811	45	1.817	1.815	56	1.809	1.808	58	1.798	1.801	48	1.794	1.797	43
0 3 4		1.811			1.816			1.806			1.796			1.790	
4 0 2	1.771	1.771	7	1.775	1.777	5	1.766	1.764	6	1.751	1.754	6	1.749	1.750	5
3 2 3		1.770			1.776			1.764			1.753			1.748	
1 3 4	1.742	1.745	14	1.747	1.750	15	1.739	1.739	18	1.728	1.729	13	1.722	1.723	13
2 4 3		1.741			1.746			1.736			1.727			1.722	
4 3 1	1.722	1.724	10	1.726	1.729	9	1.719	1.719	11	1.710	1.711	11	1.706	1.709	8
0 6 2		1.719			1.723			1.716			1.712			1.707	
1 5 3	1.694	1.695	10	1.698	1.700	8	1.690	1.691	9	1.681	1.684	8	1.678	1.679	7
$\bar{2}$ 3 4		1.692			1.697			1.690			1.681			1.677	
1 6 2	1.672	1.672	8	1.676	1.676	6	1.670	1.668	7	1.662	1.664	7	1.657	1.659	6
$\bar{4}$ 1 3		1.670			1.674			1.668			1.660			1.658	
2 3 4	1.615	1.617	6	1.620	1.622	6	1.612	1.612	4	1.603	1.601	4	1.600	1.596	4
1 4 4		1.616			1.621			1.612			1.603			1.598	
$\bar{5}$ 0 1		1.612			1.617			1.609			1.602			1.600	
1 0 5	1.595	1.596	9	1.603	1.601	8	1.590	1.590	8	1.580	1.578	8	1.575	1.573	7
3 0 4	1.587	1.587	14	1.592	1.592	10	1.582	1.581	10	1.570	1.570	10	1.567	1.565	10
0 2 5		1.585			1.589			1.580			1.569			1.565	
3 4 3	1.556	1.557	7	1.561	1.562	7	1.551	1.552	8	1.452	1.544	9	1.540	1.540	7
5 1 1		1.555			1.560			1.551			1.543			1.540	
3 4 2	1.527	1.528	13	1.530	1.534	14	1.524	1.523	12	1.515	1.513	12	1.511	1.508	12
0 5 4		1.526			1.530			1.523			1.516			1.511	

TABLE III

Atomic Parameters for Er2(CrO4)2(OH)2

UEQ = ($\frac{1}{3}$) · SUM(UIJ · AI* · AJ* · AI · AJ · COS(AI, AJ)) · 10**4

Atom	X/A	Y/B	Z/C	UEQ
ER1	0.42063(4)	0.42530(3)	0.17951(4)	60(1)
ER2	0.86755(4)	0.44317(3)	0.15250(4)	62(1)
CR1	0.63341(17)	0.17366(12)	−0.00288(16)	73(3)
CR2	0.63823(17)	0.31126(12)	0.53838(16)	74(3)
O1	0.65558(76)	0.31535(55)	0.06054(82)	102(16)
O2	0.48385(85)	0.17095(61)	−0.14716(81)	129(17)
O3	0.79771(84)	0.12769(63)	−0.08339(84)	140(18)
O4	0.59907(94)	0.08692(62)	0.15255(88)	147(18)
O5	0.65099(80)	0.38234(62)	0.36088(77)	112(17)
O6	0.51050(86)	0.20028(62)	0.51033(88)	142(17)
O7	0.81621(85)	0.25912(61)	0.61331(91)	149(18)
O8	0.57421(83)	0.40294(62)	0.67404(85)	128(17)
O9	0.62907(79)	0.55106(55)	0.09711(76)	80(15)
O10	0.85584(78)	0.49347(57)	−0.12697(78)	97(16)
H9	0.59(2)	0.57(2)	0.18(2)	150(0)
H10	0.89(3)	0.42(2)	−0.21(3)	150(0)

of the oxygen atoms belong to five different CrO$_4$ groups, and the remaining three, which are hydroxyl oxygens, bridge Er atoms.

The quadrangular antiprisms share the O(1)–O(5)–O(9) triangular faces forming couples of Er(1) and Er(2). These are linked by O(10) atoms such that chains run along the a direction (Fig. 2) (22). These chains are joined by sharing vertexes of the CrO$_4$ tetrahedra forming channels or tunnels along the same a direction.

The channels are connected between them through hydroxyl oxygen, O(9) and O(10), along the c direction. These hydroxyl groups form hydrogen bonds. One of them, (O(10), is attached to the O(6) and O(4) atoms through an asymmetrically bifurcated hydrogen bond, and the O(9) atoms are connected to O(8) through a simple hydrogen bond. There is some difference between the Er–O and the Er–OH distances, whose average values are 2.398 and 2.352 Å, respectively.

Geometrical features of the hydrogen bonds are specified at the end of Table IV.

Nevertheless, this three-dimensional structure of Er(OH)CrO$_4$ can be viewed as being formed by parallel slices of alternating Er polyhedra and CrO$_4$ tetrahedra sharing vertices perpendicular to the b axis.

On comparing this new structure with those corresponding to Ln(OH)CrO$_4$ (15) (Ln = La, Pr, Nd) some differences can be observed: In La(OH)CrO$_4$ the La atom is coordinated to nine oxygen atoms in a very irregular manner, whereas in Er(OH)CrO$_4$ the Er occur in an almost quadrangular antiprism exhibiting a lower coordination, as expected on the basis of the differences in the ionic radii between La(III) and Er(III). The coordination distances in ErO$_8$ are shorter (average 2.379 Å) than LaO$_9$ (average 2.578 Å).

On the other hand, in (15) the Ln polyhedra are shown as sharing vertexes, although in the Er series polyhedra couples share faces; thus, the structure is more compact, in spite of the existence of tunnels. This fact agrees with their density values of 4.41 g · cm^{-3} and 5.28 g · cm^{-3}, respectively.

TABLE IV

INTERATOMIC BOND DISTANCES (Å) AND ANGLES (°) IN Er₂(CrO₄)₂(OH)₂

ER(1)–O(1)	2.533(6)	ER(2)–O(2)[5]	2.251(7)
ER(1)–O(5)	2.358(6)	ER(2)–O(4)[4]	2.290(7)
ER(1)–O(9)	2.348(6)	ER(2)–O(6)[6]	2.357(7)
ER(1)–O(3)[7]	2.337(7)	ER(2)–O(10)[1]	2.378(6)
ER(1)–O(7)[8]	2.304(7)	CR(1)–O(1)	1.692(6)
ER(1)–O(8)[2]	2.289(7)	CR(1)–O(2)	1.637(7)
ER(1)–O(9)[3]	2.304(6)	CR(1)–O(3)	1.617(7)
ER(1)–O(10)[3]	2.432(6)	CR(1)–O(4)	1.655(7)
ER(2)–O(1)	2.332(6)	CR(2)–O(5)	1.680(7)
ER(2)–O(5)	2.636(7)	CR(2)–O(6)	1.634(7)
ER(2)–O(9)	2.305(6)	CR(2)–O(7)	1.638(7)
ER(2)–O(10)	2.371(6)	CR(2)–O(8)	1.637(7)

O(9)[3]–ER(1)–O(10)[3]	71.7(2)	O(9)–ER(2)–O(10)[1]	127.3(2)
O(8)[2]–ER(1)–O(10)[3]	75.8(2)	O(9)–ER(2)–O(6)[6]	134.6(2)
O(8)[2]–ER(1)–O(9)[3]	114.8(2)	O(9)–ER(2)–O(4)[4]	79.5(2)
O(7)[8]–ER(1)–O(10)[3]	89.0(2)	O(9)–ER(2)–O(2)[5]	138.7(2)
O(7)[8]–ER(1)–O(9)[3]	80.5(2)	O(9)–ER(2)–O(10)	72.9(2)
O(7)[8]–ER(1)–O(8)[2]	152.6(2)	O(5)–ER(2)–O(10)[1]	144.4(2)
O(3)[7]–ER(1)–O(10)[3]	78.6(2)	O(5)–ER(2)–O(6)[6]	121.2(2)
O(3)[7]–ER(1)–O(9)[3]	143.5(2)	O(5)–ER(2)–O(4)[4]	77.2(2)
O(3)[7]–ER(1)–O(8)[2]	76.6(2)	O(5)–ER(2)–O(2)[5]	68.5(2)
O(3)[7]–ER(1)–O(7)[8]	78.1(2)	O(5)–ER(2)–O(10)	135.7(2)
O(9)–ER(1)–O(10)[3]	113.1(2)	O(5)–ER(2)–O(9)	71.3(2)
O(9)–ER(1)–O(9)[3]	73.8(2)	O(1)–ER(2)–O(10)[1]	146.7(2)
O(9)–ER(1)–O(8)[2]	69.6(2)	O(1)–ER(2)–O(6)[6]	77.7(2)
O(9)–ER(1)–O(7)[8]	137.8(2)	O(1)–ER(2)–O(4)[4]	135.8(2)
O(9)–ER(1)–O(3)[7]	139.2(2)	O(1)–ER(2)–O(2)[5]	98.1(2)
O(5)–ER(1)–O(10)[3]	150.3(2)	O(1)–ER(2)–O(10)	81.7(2)
O(5)–ER(1)–O(9)[3]	136.6(2)	O(1)–ER(2)–O(9)	71.1(2)
O(5)–ER(1)–O(8)[2]	81.8(2)	O(1)–ER(2)–O(5)	62.6(2)
O(5)–ER(1)–O(7)[8]	102.9(2)	O(3)–CR(1)–O(4)	108.4(4)
O(5)–ER(1)–O(3)[7]	77.4(2)	O(2)–CR(1)–O(4)	113.3(4)
O(5)–ER(1)–O(9)	75.8(2)	O(2)–CR(1)–O(3)	106.5(3)
O(1)–ER(1)–O(10)[3]	145.7(2)	O(1)–CR(1)–O(4)	110.1(3)
O(1)–ER(1)–O(9)[3]	75.9(2)	O(1)–CR(1)–O(3)	111.1(3)
O(1)–ER(1)–O(8)[2]	129.6(2)	O(1)–CR(1)–O(2)	107.4(3)
O(1)–ER(1)–O(7)[8]	74.0(2)	O(7)–CR(2)–O(8)	106.5(3)
O(1)–ER(1)–O(3)[7]	125.2(2)	O(6)–CR(2)–O(8)	110.8(4)
O(1)–ER(1)–O(9)	67.0(2)	O(6)–CR(2)–O(7)	107.9(3)
O(1)–ER(1)–O(5)	64.0(2)	O(5)–CR(2)–O(8)	109.6(3)
O(6)[6]–ER(2)–O(10)[1]	70.3(2)	O(5)–CR(2)–O(7)	113.2(3)
O(4)[4]–ER(2)–O(10)[1]	77.5(2)	O(5)–CR(2)–O(6)	108.8(3)
O(4)[4]–ER(2)–O(6)[6]	143.3(2)	ER(2)–O(1)–CR(1)	138.5(3)
O(2)[5]–ER(2)–O(10)[1]	83.7(2)	ER(1)–O(1)–CR(1)	121.3(3)
O(2)[5]–ER(2)–O(6)[6]	77.0(2)	ER(1)–O(1)–ER(2)	97.2
O(2)[5]–ER(2)–O(4)[4]	82.6(2)	ER(2)–O(5)–CR(2)	141.2(3)
O(10)–ER(2)–O(10)[1]	79.2(2)	ER(1)–O(5)–CR(2)	123.8(3)
O(10)–ER(2)–O(6)[6]	70.6(2)	ER(1)–O(5)–ER(2)	93.8
O(10)–ER(2)–O(4)[4]	120.4(2)	ER(1)–O(9)–ER(2)	103.4(2)
O(10)–ER(2)–O(2)[5]	146.9(2)		

X–H . . . O	X–H	H . . . O	X . . . O	X–H . . . O
O(9) –H(9) . . O(8)[2]	0.82(19)	1.88(19)	2.65(1)	155.(18)
O(10)–H(10) . . O(4)[6]	1.09(22)	2.12(22)	2.92(1)	128.(15)
O(10)–H(10) . . O(6)[6]	1.09(22)	2.44(21)	2.732(9)	93.(12)

Note. Symmetry transformations: (1) $1 + (1 - x)$, $(1 - y)$, $(-z)$; (2) $(1 - x)$, $(1 - y)$, $(1 - z)$; (3) $(1 - x)$, $(1 - y)$, $(-z)$; (4) $1 + (1/2 - x)$, $(1/2 + y)$, $(1/2 - z)$; (5) $(x + 1/2)$, $(1/2 - y)$, $(1/2 + z)$; (6) $(x + 1/2)$, $(1/2 - y)$, $(z - 1/2)$; (7) $(x - 1/2)$, $(1/2 - y)$, $(1/2 + z)$; (8) $(x - 1/2)$, $(1/2 - y)$, $(z - 1/2)$.
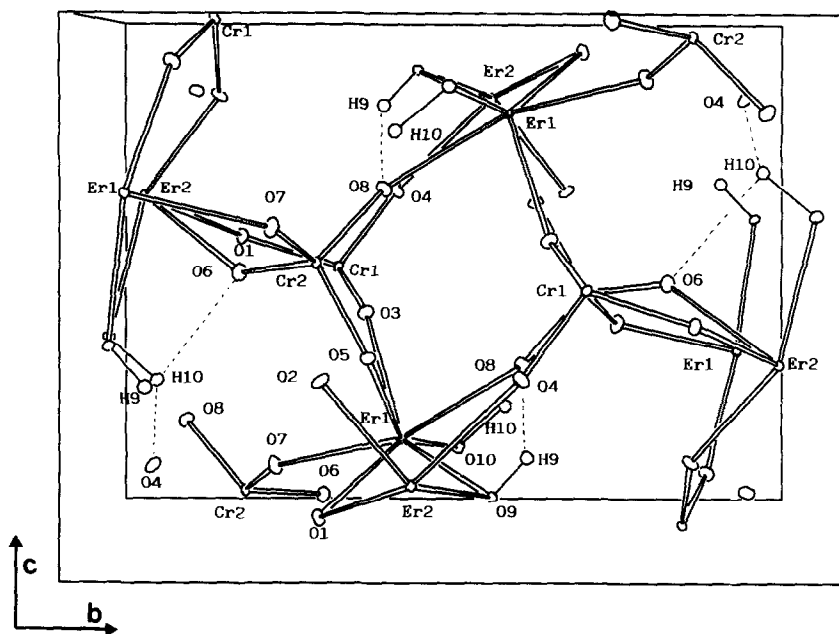
FIG. 2. A view of the unit cell of $Er(CrO_4)OH$.

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