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The Crystal Structure of α-*Cis*-di-μ-hydroxobis(ethylenediamine-N,N'-diacetato)-dichromium(III) Tetrahydrate, $[Cr(EDDA)(OH)]_2 \cdot 4H_2O$

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The crystal and molecular structure of α-cis*-di-μ-hydroxobis(ethylenediamine-N,N'-diacetato)dichromium (III) tetrahydrate,* $[Cr(EDDA)(OH)]_2 \cdot 4H_2O$, *was determined from three-dimensional X-ray diffractometer data. The complex crystallizes in monoclinic system, space group* $P2_1/n$, *with* a = *11.831(5),* b = *16.409(8),* c = *5.419(3)* Å, *β = 99.05(4)°, Z = 2. The structure was solved by the heavy atom method and atomic parameters refined by full-matrix least-squares methods to final R = 0.048 for 1389 independent reflections having I ⩾ 3σ(I). The structure consists of dimeric* $[Cr(EDDA)(OH)]_2$ *molecules and molecules of water. The geometry around a chromium atom is roughly octahedral, with tetradentate EDDA (EDDA = ethylenediamine-N,N'-diacetate ion) assuming α-*cis *configuration in which the complex possesses two out-of-plane glycinate (R) rings, and the backbone ethylenediamine (E) ring. The bridging unit is strictly planar, there being an inversion centre in the middle of the dimer. The Cr–O–Cr bridging angle is 97.6(1)° and Cr–Cr separation 2.950(2) Å. Comparison of the sums of the bond angles (as an estimate of ring strains (1)) in* $[Cr(EDDA)(OH)]_2$ *molecule and related Co(III) complexes indicates that the effect of configuration is a more important factor than the size of metal ion.*

Introduction

The structural data of the $[Co(EDTA)]^-$ complex [1] and closely related chelate system [2] show the difference in planarity of the G (in-plane) and R (out-of-plane) glycinate rings. The G rings of these systems are more strained relative to the R glycinate rings. Due to the structural similarity between the EDDA and EDTA ligands, the same designations of the rings have been used recently in a crystallographic study of an EDDA–Co(III) chelate [3]. It was suggested that the observed strain of the G ring [3] might be an important factor in formation of the favoured α-*cis* configuration of chelated EDDA in Co(III) complexes [4, 5].

Recently, several octahedral $[Cr(N)_2(O)_4]$ type complexes containing the EDDA chelate system have been synthetized and characterized [6]. Due to the presence of a larger metal ion, the strain of the G rings might be expected to be a stronger contributing factor in determining the configuration of Cr(III) complexes than in Co(III) complexes.

In order to confirm the configuration of chelated EDDA in the prepared $[Cr(N)_2(O)_4]$ type complexes, the X-ray crystal structure analysis of the di-μ-hydroxobis(ethylenediamine-N,N'-diacetato)dichromium(III) tetrahydrate, $[Cr(EDDA)(OH)]_2 \cdot 4H_2O$ was undertaken.

Experimental

The title compound has been prepared using the procedure given by Radanović *et al.* [6]. By a gradual addition of sodium hydroxide to the water solution (60°) containing the monomeric diaqua-EDDA-Cr(III) complex the blue-violet neutral complex precipitated. Crystals suitable for collecting the data were recrystallized from water solution by slow cooling. The prismatic crystals belong to monoclinic system. Systematic absences were observed for reflections 0k0 with k odd and h0l with h + l odd, which uniquely determine the space group as $P2_1/n$. The unit cell parameters were initially obtained from rotation and Weissenberg photographs and later adjusted by the least-squares treatment of the setting angles of 15 reflections centred on a Syntex $P\bar{1}$ four circle diffractometer, using Mo-Kα radiation. They are: *a* = 11.831(5), *b* = 16.409(8), *c* = 5.419(3) Å, β = 99.05(4)°, V = 1039(1) Å^3. The formula weight is 558.4 ($Cr_2C_{12}H_{30}O_{14}N_4$). Density, measured by flotation in bromoform–carbon tetrachloride is 1.75 g cm^{-3}; D_x = 1.78 g cm^{-3}, calculated for Z = 2, corresponds to a half formula unit (*i.e.* a half complex

TABLE I. Final Atomic Coordinates with Their e.s.d.'s. The values have been multiplied by 10^4 for the non-hydrogen atoms and by 10^2 for the hydrogen atoms.

Atom	x	y	z
Cr	4024.6(5)	5514.3(4)	412(1)
O(1)	4343(3)	4596(2)	−1700(5)
O(2)	3533(2)	4830(2)	3065(5)
O(3)	2040(3)	4256(2)	4405(6)
O(4)	4308(3)	6333(2)	−2089(5)
O(5)	4783(4)	7616(4)	−2618(6)
O(W1)	4679(4)	875(3)	297(9)
O(W2)	2863(3)	3194(4)	−1390(10)
N(1)	2260(3)	5589(2)	−790(6)
N(2)	3971(3)	6551(2)	2456(6)
C(1)	2456(4)	4648(3)	2830(8)
C(2)	1708(4)	4937(3)	482(9)
C(3)	1901(4)	6423(3)	−176(9)
C(4)	2537(4)	6671(3)	2387(9)
C(5)	4369(5)	7233(3)	1406(9)
C(6)	4500(4)	7073(3)	−1320(2)
H(1)	9.5(6)	51.1(4)	8(1)
H(2)	15.6(6)	45.2(4)	94(1)
H(3)	20.7(5)	55.1(4)	76(1)
H(4)	10.0(6)	64.8(4)	97(1)
H(5)	20.3(6)	68.1(4)	84(1)
H(6)	22.7(6)	63.5(4)	37(1)
H(7)	24.1(6)	71.7(4)	29(1)
H(8)	41.1(6)	65.2(4)	39(1)
H(9)	51.0(6)	73.3(4)	24(1)
H(10)	40.9(6)	77.3(4)	18(1)
H(11)	43.0(7)	45.8(4)	71(1)
H(12)	52.2(7)	7.8(5)	100(1)
H(13)	47.8(8)	13.2(5)	7(2)

molecule and two water molecules) per asymmetric unit. μ(Mo-Kα) = 11.8 cm^{-1}.

The intensity data were collected from a crystal of dimensions 0.11 × 0.14 × 0.30 mm mounted on a Syntex P$\bar{1}$ diffractometer with graphite-monochromated Mo-Kα radiation. The ω scanning mode and a variable scan speed were used. One standard reflection was monitored every 30 reflections and showed no systematic decrease in intensity. A unique set of 3383 intensities having $2\theta \leqslant 60°$ was collected. 1839 reflections were found to have intensities greater than 3 times their standard deviations. They were considered as observed and used in the subsequent structure analysis and refinement. The values of I and σ(I) were corrected for Lorentz and polarization effects. Absorption correction was not applied.

Determination and Refinement of the Structure

The position of chromium atom was deduced from a three-dimensional Patterson function, and the remaining non-hydrogen atoms were located after subsequent least-squares iterations and difference Fourier summations. Isotropic least-squares refinement of these atoms gave R = 0.09 and R_w = 0.14. Anisotropic refinement reduced these values to 0.06 and 0.09 respectively. A difference electron density map calculated at this point revealed the positions of all hydrogen atoms, except those belonging to one of water molecules. The final least-squares cycles involved anisotropic refinement of non-hydrogen atoms and isotropic refinement of hydrogen coordinates with thermal parameters fixed. Refinement converged at R = 0.048 and R_w = 0.058. All the least-squares refinements in this analysis were carried out on F, the function minimized being $\Sigma w(|F_o| - |F_c|)^2$; the weights w were taken as $1/\sigma^2$. The final difference Fourier map had no peaks higher than 0.56 e Å^{-3}. The final parameter shifts of non-hydrogen atoms were below 0.3σ. Scattering factors for all atoms, as well as the anomalous dispersion correction for chromium atom were taken from ref. [7].

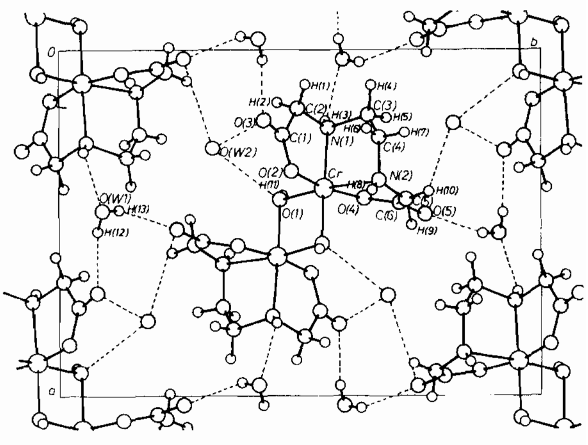

Fig. 1. The c-axis projection of the crystal structure of $[Cr(EDDA)(OH)]_2 \cdot 4H_2O$.

All calculations were performed on the CDC-3600 computer using the programmes FORDAP (Zalkin's Fourier programme), NUCLS (least-squares, J. A. Ibers) and GEOM (molecular geometry calculations, K. W. Muir and P. Mallinson). The positional and thermal parameters deriving from the final least-squares calculation along with their standard deviations, as estimated from the inverse matrix, are presented in Tables I and II. A list of the observed and calculated structure amplitudes is given in a Table available from the Editor.

Discussion

The structure consists of dimeric [Cr(EDDA)-(OH)]$_2$ molecules and molecules of water, linked

TABLE II. Final Thermal Parameters with Their e.s.d.'s. Anisotropic thermal parameters are in the form: $\exp(-\sum_i\sum_j \beta_{ij}h_ih_j)$. The values are multiplied by 10^4.

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Cr	24.7(4)	15.8(2)	9.9(2)	−0.3(3)	9.3(6)	−0.2(7)
O(1)	29(2)	18(1)	119(9)	−1(1)	4(4)	1(3)
O(2)	28(2)	24(1)	128(9)	−4(1)	10(3)	8(3)
O(3)	42(3)	44(2)	279(13)	−7(2)	23(5)	50(4)
O(4)	44(2)	19(1)	111(9)	−2(1)	16(4)	2(2)
O(5)	118(4)	24(1)	247(13)	−23(2)	78(6)	−1(3)
O(W1)	58(3)	26(1)	382(17)	−3(2)	51(6)	−14(5)
O(W2)	163(7)	68(3)	986(38)	−11(4)	10(14)	38(9)
N(1)	33(2)	23(1)	108(11)	0(2)	5(4)	4(3)
N(2)	45(3)	18(1)	126(11)	−2(2)	27(5)	−1(3)
C(1)	38(3)	21(2)	165(14)	−3(2)	6(5)	3(4)
C(2)	34(3)	36(2)	185(17)	−7(2)	−3(6)	8(5)
C(3)	40(3)	26(2)	231(18)	11(2)	20(6)	3(4)
C(4)	49(4)	25(2)	212(17)	8(2)	30(6)	−14(4)
C(5)	74(5)	23(2)	166(16)	−12(2)	35(7)	−19(4)
C(6)	53(4)	24(2)	149(14)	−5(2)	17(6)	−2(4)

TABLE III. Interatomic-Distances (A) and Bond Angles (°) with Their e.s.d.'s. The superscript marks the position: $\bar{x}\ \bar{y}\ \bar{z}$.

Cr–Cr^1	2.950(1)	Cr–O(1)^1	1.956(3)
Cr–O(1)	1.964(3)	O(5)–C(6)	1.215(5)
Cr–O(2)	1.982(3)	N(1)–C(2)	1.479(6)
Cr–O(4)	1.974(3)	N(1)–C(3)	1.486(6)
Cr–N(1)	2.090(4)	N(2)–C(4)	1.491(6)
Cr–N(2)	2.072(3)	N(2)–C(5)	1.472(6)
O(2)–C(1)	1.295(5)	C(1)–C(2)	1.508(7)
O(3)–C(1)	1.232(6)	C(3)–C(4)	1.527(7)
O(4)–C(6)	1.292(6)	C(5)–C(6)	1.532(5)
O(2)–Cr–O(1)	95.3(1)	O(4)–Cr–O(1)	93.0(1)
N(1)–Cr–O(1)	97.9(1)	N(2)–Cr–O(1)	174.4(1)
O(4)–Cr–O(2)	169.9(1)	N(1)–Cr–O(2)	82.3(1)
N(2)–Cr–O(2)	89.9(1)	C(1)–O(2)–Cr	117.1(2)
N(1)–Cr–O(4)	90.9(1)	N(2)–Cr–O(4)	81.9(1)
C(6)–O(4)–Cr	117.2(2)	N(2)–Cr–N(1)	84.7(1)
C(2)–N(1)–Cr	107.8(3)	C(3)–N(1)–Cr	106.9(3)
C(4)–N(2)–Cr	108.1(2)	C(5)–N(2)–Cr	108.0(3)
O(3)–C(1)–O(2)	123.1(4)	C(2)–C(1)–O(2)	116.6(4)
C(2)–C(1)–O(3)	120.2(4)	O(5)–C(6)–O(4)	123.5(2)
C(5)–C(6)–O(4)	115.6(3)	C(5)–C(6)–O(5)	120.8(4)
C(3)–N(1)–C(2)	113.5(3)	C(1)–C(2)–N(1)	112.2(4)
C(4)–C(3)–N(1)	109.4(4)	C(5)–N(2)–C(4)	114.3(4)
C(3)–C(4)–N(2)	109.5(4)	C(6)–C(5)–N(2)	111.5(4)
O(1)^1–Cr–O(1)	82.4(1)	O(1)^1–Cr–N(2)	95.3(1)
O(1)^1–Cr–O(2)	94.3(1)	O(1)^1–Cr–O(4)	92.5(1)
O(1)^1–Cr–N(1)	176.6(1)		

together by hydrogen bonds. A view of the structure down the *c*-axis is shown in Fig. 1. The centre of the dimer is constrained to lie on the inversion centre. The geometry around chromium atom is approximately octahedral, the ligating atoms being two O and two N atoms from EDDA ligand and two O atoms from the two hydroxo-bridging groups. The bond lengths and angles in the inner coordination sphere are given in Table III. The EDDA ligand is tetradentate. Coordinating with the chromium ion it forms three five-membered rings: one ethylenediamine ring, which lies in the plane of the two co-

TABLE IV. Torsion Angles in Five-membered Chelate R and E Rings in $[Cr(EDDA)(OH)]_2$.

Cr–O(2)–C(1)–C(2)	3.5(5)
O(2)–C(1)–C(2)–N(1)	–17.5(6)
Cr–N(1)–C(2)–C(1)	21.4(4)
O(2)–Cr–N(1)–C(2)	–15.7(3)
N(1)–Cr–O(2)–C(1)	7.3(3)
Cr–O(4)–C(6)–C(5)	–5.6(5)
N(2)–C(5)–C(6)–O(4)	–13.1(5)
Cr–N(2)–C(5)–C(6)	23.6(4)
O(4)–Cr–N(2)–C(5)	–21.0(3)
N(2)–Cr–O(4)–C(6)	15.5(3)
Cr–N(1)–C(3)–C(4)	–40.2(4)
N(1)–C(3)–C(4)–N(2)	52.2(5)
Cr–N(2)–C(4)–C(3)	–36.5(4)
N(1)–Cr–N(2)–C(4)	11.4(3)
N(2)–Cr–N(1)–C(3)	16.0(3)

TABLE VI. Weighted Least-squares Planes and Distances (A) of Atoms from These Planes.

Plane I:	C(1), C(2), O(2), O(3)		
Eq:	0.2509x – 0.8516y – 0.4603z – 6.5216 = 0		
C(1)	–0.0022	O(3)	0.0004
C(2)	0.0007	N(1)	–0.4060
O(2)	0.0003	Cr	–0.0997
Plane II:	C(5), C(6), O(4), O(5)		
Eq:	0.9285x – 0.2280y + 0.2930z + 2.1995 = 0		
C(5)	0.0025	O(5)	0.0018
C(6)	–0.0052	N(2)	–0.2958
O(4)	0.0011	Cr	0.1902

ordinated nitrogen atoms (E ring) and two glycinate rings, lying outside this plane (R rings). Hence, the configuration is *α-cis*, as predicted on the basis of absorption spectroscopy data [6]. The crystallographic symmetry of the molecule is $C_i(\bar{1})$, but it approximates C_{2h} (2/m); in this respect it is similar to all the previously reported hydroxo-bridged chromium(III) dimers [8, 12], except $[Cr(phen)_2(OH)]_2^{4+}$ cation [13, 14], which approximates D_2 (222) symmetry. The two Cr–O distances in the bridging unit are 1.964(3) and 1.956(3) Å. These are close to values found in other μ-hydroxo dimer complexes which are in the range from 1.949 to 1.987 Å, with exception of somewhat shorter Cr–O distances in $[Cr(phen)_2(OH)]_2^{4+}$ cation (1.927 Å). Owing to the inversion centre, the Cr–O–Cr–O bridging unit is strictly planar, the Cr–O–Cr angle being 97.6(1)° and the Cr–Cr separation 2.950(2) Å. Both values are the smallest of all the values found in μ-hydroxo-bridged Cr(III) dimers, which are in the range from 98.2–103.4° and 2.974–3.059 Å.

The geometry of ethylenediamine and the two glycinate chelate rings is normal; the bond lengths and angles are in the range found in similar compounds [1, 2, 3], but also in the complexes where the chelate rings are not fused [8, 9, 15–20].

The conformation of the chelate rings is such that both glycinate R rings have the same conformation, while the ethylenediamine E ring has an opposite conformation. The presence of the inversion centre, of course, constrains each dimer to contain an equal number of λ and δ rings, as well as chromium atoms in the dimer to have opposite configurations. Chromium atom which can be described as a skew pair Δ has the configuration Δ (λ δ λ) [21]. consequently, the chromium atom related by the inversion centre has Λ (δ λ δ) configuration. Absolute configurations of two asymmetric nitrogen atoms are SS and RR in the Λ and Δ chirality, respectively. The two glycinate chelate rings have an envelope conformation. The ring carbon atoms C(2) and C(1) lie 0.38 and 0.14 Å respectively below the plane formed by N(1), Cr and O(2). The corresponding deviations of C(6) and C(5) atoms from the plane of O(4), Cr, N(2) are 0.30 and 0.50 Å. In the central, ethylenediamine chelate ring, C(4) and C(3) are found to lie at 0.28 and –0.39 Å from the plane of N(2), Cr, N(1), respectively. The torsion angles in the rings are given in Table IV. They were defined as usually: for atoms A, B, C and D bonded in chain, the torsion angle about the bond BC was taken as positive if the bond AB was required to be rotated clockwise to lie in the BCD plane. For the O–C–C–N and N–C–C–N chains of the chelate ring the method would assign a positive sign to the torsion angle

TABLE V. Bond Angle Sums in $[Co(EDTA)]^-$, $[Co(EDDA)(R\text{-}pn)]^+$ and $[Cr(EDDA)(OH)]_2$.

Ring	Ideal	$[Co(EDTA)]^-$	$[Co(EDDA)(R\text{-}pn)]^+$	$[Cr(EDDA)(OH)]_2$
R	538.4	537.8	532.5	537.0 534.3
E	527.9	520.9	517.7	518.6
G	538.4	523.5	529.0	

TABLE VII. Probable Hydrogen Bonds.

A–H. . .B	A–B	H. . .B	<A–H. . .B	<B. . .A. . .B
N(1)–H(3). . .O(W1)^1	3.112	2.485	134.0	
N(2)–H(8). . .O(4)^2	2.945	2.221	158.6	
O(1)–H(11). . .O(2)^5	2.873	2.301	169.0	
O(W1)–H(12). . .O(3)^4	2.917	2.182	158.7	
O(W1)–H(13). . .O(5)^5	2.805	2.376	131.5	
O(W2). . .O(1)	2.912			67.2 142.9
O(W2). . .O(3)^3	2.912			81.4
O(W2). . .O(5)^6	3.234			

Symmetry Code

1	½ – x	½ + y	–z – ½
2	x	y	1 + z
3	x	y	z – 1
4	½ + x	½ – y	z – 12
5	1 – x	1 – y	–z
6	½ – x	y – ½	–z – ½

about the C–C bond when the conformation is δ and a negative sign for λ conformation. The value for dihedral angles about C–C bonds for the two R glycinate rings belonging to Cr with Δ configuration, are –17.5 and –13.1°. The X-ray structural studies have shown that these angles are between 0 and 30°, in accordance with energy minimization calculations [22]. In the E ring, the torsion N–C–C–N angle is 52.2°. In a number of ethylenediamine complexes this value is also about 50°.

Displacement of the corresponding atoms from the best least-squares planes through carboxylato groups shows that they are planar (Table VI). In the ring containing C(1), C(2), O(2), O(3) group, Cr and N are displaced on the same side of the plane, while in the second R ring Cr and N lie on opposite sides of the C(5), C(6), O(4), O(5) plane. The planes of the two R and E rings are mutually perpendicular, the angles between R rings being 90.3 and between R and E 87.8 and 88.0°.

Weakliem and Hoard [1] suggested that the sum of bond angles of the rings could be used as an estimate of ring strain. For the five-membered glycinate rings, the ideal bond angle sum is 538.4°, which allows the rings to be nearly planar. The ideal value for the sum of the bond angles of the five-membered ethylenediamine E ring is 527.9°. Table V compares the bond angle sums for the chelate rings in [Co(EDTA)]$^-$, [Co(EDDA)(R-pn)]$^+$ and [Cr(EDDA)(OH)]$_2$. Chelated EDTA possesses the full ringsystem and the ring strains are mostly concentrated in G rings [1]. In [Co(EDDA)(R-pn)]$^+$ ion the configuration is *β-cis*; EDDA forms R, F and G rings, which are more strained than in EDTA complex [3]. [Cr(EDDA)(OH)]$_2$ has *α-cis* configuration, but it also possesses a slightly larger metal ion. The sums of the bond angles in R rings are close to the ideal value, the ring strains being more pronounced in E ring. The values are, however, lower than those in EDDA-Co(III) complex, indicating that in determining the ring strains the favoured *α-cis* configuration is a more important factor than the size of the metal ion.

The crystal packing incorporates a considerable network of hydrogen bonds linking dimeric molecules and molecules of water. Table VII lists the parameters associated with hydrogen bonds that are suggested by the usual criteria [23]. Inspection of Table VII indicates that hydroxo oxygen and N(2) amino nitrogen atoms are involved in hydrogen bonds linking the dimers in the direction of *c* axis. Water molecule O(W1) is a donor for two hydrogen bonds with carboxyl oxygen atoms from the two adjacent dimeric molecules; it is also acceptor for N(1)–H(3). . .O(W1) hydrogen bond from the third dimeric molecule. The inability to locate the hydrogen atoms on O(W2) precludes any serious discussion on hydrogen bonding for this molecule. However, according to the O(W2). . .O separations and corresponding O. . .O(W2). . .O angles it seems that O(W2) forms one normal and one bifurcated bond, linking also three adjacent molecules of dimers.

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