

Table 4. Hydrogen-bond distances and angles in myo-inositol-2-phosphate monohydrate

<i>i</i>	<i>j</i>	<i>k</i>	<i>d</i> _{O...O}	<i>d</i> _{O...H}	∠ _{<i>ijk</i>}	Symmetry operation of <i>k</i> *
O(1)——H(O1)·····O(4)	2.684 Å	1.82 Å	166°	455·1		
O(1)·····H(OX3)——OX(3)	2.603	1.82	159	454·3		
O(3)——H(O3)·····OX(4)	2.733	2.00	161	756·2		
O(3)·····H(OX1)——OX(1)	2.537	1.74	173	655·1		
O(4)——H(O4)·····OW	2.716	1.95	177	655·1		
O(4)·····H(O1)——O(1)	2.684	1.82	166	655·1		
O(5)——H(O5)·····OX(3)	2.802	2.35	106	454·1		
O(5)·····H1(OW)——OW	2.815	1.95	165	655·2		
O(6)——H(O6)·····OW	2.787	1.91	175	555·2		
OX(1)——H(OX1)·····O(3)	2.537	1.74	173	455·1		
OX(3)——H(OX3)·····O(1)	2.603	1.82	159	655·3		
OX(3)·····H(O5)——O(5)	2.802	2.35	106	656·1		
OX(4)·····H2(OW)——OW	2.905	2.07	163	555·1		
OX(4)·····H(O3)——O(3)	2.733	2.00	161	756·2		
OW·····H(O4)——O(4)	2.717	1.95	177	455·1		
OW·····H(O6)——O(6)	2.787	1.91	175	555·2		
OW——H1(OW)·····O(5)	2.815	1.95	165	655·2		
OW——H2(OW)·····OX(4)	2.905	2.07	163	555·1		

* Reference point is atom *i* at 555·1. The first three digits code a lattice translation, e.g. 466·1 is $-a+b+c$ from 555·1. The last digit indicates one of the following operations:

- 1: x, y, z 2: $\bar{x}, \bar{y}, \bar{z}$
 3: $x, \frac{1}{2}-y, \frac{1}{2}+z$

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The Crystal and Molecular Structure of Diaquo- μ -triethylenetetraminehexaacetatodichromium(III) hexahydrate, $[\text{Cr}_2\text{TTHA} \cdot 2\text{H}_2\text{O}] \cdot 6\text{H}_2\text{O}$

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The title compound crystallizes in space group $P2_1/c$ with $a = 8.634$ (5), $b = 12.606$ (6), $c = 13.867$ (7) Å, $\beta = 99.67$ (8)°, $D_m = 1.63$ (1), $D_c = 1.64$ g cm⁻³ for $Z = 2$. The structure has been determined by direct and Fourier methods, refined to $R = 0.043$ from diffractometer data (1952 observed reflexions), and consists of discrete binuclear units which are required crystallographically to be centrosymmetric. The TTHA ion (triethylenetetraminehexaacetate) acts as a pentadentate ligand to each of the chromium atoms, the octahedra of which are completed by water molecules. The bond distances about each chromium are Cr–N, 2.120 and 2.042; Cr–OH₂, 1.997; Cr–O, 1.946, 1.959 and 1.943 Å.

Introduction

In the last decade many structural studies have been carried out on the coordinating ability of the potential hexadentate ligand EDTA (Richards, Pedersen, Silver-

ton & Hoard, 1964; Lin, Leggett & Wing, 1973; Hamor, Hamor & Hoard, 1964). It has been found that EDTA can act as a pentadentate ligand, one $-\text{CH}_2\text{COOH}$ arm being free (Stephens, 1969; Smith & Hoard, 1959), as a hexadentate ligand in six- (Weakliem &

Hoard, 1959; Porai-Koshits, Novashilova, Polynova, Filippova & Martynenko, 1973) or seven- (Lind & Hoard, 1964; Hamor, Hamor & Hoard, 1964) coordinate complexes or as a bridging ligand (Filippova, Polynova, Porai-Koshits, Novashilova & Martynenko, 1973; Park, Glick & Hoard, 1969). The related ligand triethylenetetraminehexaacetic acid (H_6TTHA) (a ligand with ten potential coordinating sites) has been extensively used as a coordinating ligand in e.s.r. studies, from which M–M contact distances are derived and possible structures for the complex proposed (Smith & Martell, 1972). The present crystallographic analysis was undertaken to determine the configuration of the TTHA ligand and the intra- and intermolecular metal–metal separation in one such complex.

Experimental

Crystal data

$\text{C}_{18}\text{H}_{40}\text{O}_{20}\text{N}_4\text{Cr}_2$, $M = 736.6$, monoclinic, $a = 8.634(5)$, $b = 12.606(6)$, $c = 13.867(7)$ Å, $\beta = 99.67(8)^\circ$, $U = 1487.8$ Å³. $D_m = 1.63(1)$ g cm⁻³ (by flotation in carbon tetrachloride/1,1,2,2-tetrabromoethane), $Z = 2$, $D_c = 1.64$ g cm⁻³. $F(000) = 384$, $\mu = 8.9$ cm⁻¹ for Mo $K\alpha$ radiation ($\lambda = 0.7107$ Å). Space group $P2_1/c$ (No. 14) from systematic absences: $h0l$, l odd; $0k0$, k odd.

Unit-cell parameters were determined with a standard Philips PW 1100 X-ray diffractometer computer program. The program investigates rows in the reciprocal lattice through the origin and scans the four highest weighted reflexions and their anti-reflexions (weighted by intensity and $\sin \theta$). The centres of gravity of these eight profiles are then used in a least-squares refinement of the d spacing for that row.

Intensity measurements

All intensities were collected from one crystal of dimensions $0.12 \times 0.15 \times 0.13$ mm with a Philips PW 1100 diffractometer and Mo $K\alpha$ radiation. 2626 reflexions of the unique data set were collected out to 2θ (Mo $K\alpha$) = 50.0° . Of these reflexions 1952 were considered to be significantly above background [$I > 3\sigma(I)$], and only these were used in the subsequent calculations. Three standard reflexions measured at two-hourly intervals showed no significant variations in intensity.

The data were collected by the θ – 2θ scan technique with a symmetric scan range of $\pm 0.90^\circ$ in 2θ from the calculated Bragg angle, at a scan rate of $0.025^\circ\text{min}^{-1}$. The radiation was monochromatized with a flat graphite monochromator and no reflexion was sufficiently intense to require the insertion of an attenuation filter.

The data were processed with a program written specifically for the PW 1100 diffractometer (Hornstra & Stubbe, 1972). The background-corrected intensities were assigned standard deviations according to: $\sigma(I) = [CT + (t_c/t_b)^2 (B_1 + B_2) + (pI)^2]^{1/2}$ where CT is the total integrated peak count obtained in a scan time t_c ; B_1 and B_2 are background counts each obtained in time $\frac{1}{2}t_b$, and $I = CT - (t_c/t_b) (B_1 + B_2)$; p was 0.04. The inclusion of this last term is to allow for 'machine errors'. Values of I and $\sigma(I)$ were then corrected for Lorentz and polarization effects. Neither extinction nor absorption corrections were applied.

Structure determination and refinement

The structure was solved for the chromium atom position by standard heavy-atom methods. However, further progress was impeded as the chromium y/b

Table 1. Final positional parameters ($\times 10^4$) and anisotropic thermal parameters ($\times 10^3$)* of non-hydrogen atoms

Estimated standard deviations are given in parentheses. The temperature factor is of the form

$$\exp[-2\pi^2(U_{11}h^2a^{*2} + \dots + 2U_{23}b^*c^*kl + \dots)].$$

	x/a	y/b	z/c	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Cr	1572 (1)	2356 (1)	3880 (1)	280 (6)	262 (6)	252 (6)	-58 (6)	42 (5)	-7 (6)
O(1)	5594 (8)	776 (7)	4259 (6)	37 (4)	86 (6)	107 (7)	18 (4)	11 (4)	-22 (5)
O(2)	3135 (6)	1273 (5)	3762 (4)	30 (3)	41 (4)	44 (4)	-1 (3)	11 (3)	-9 (3)
O(3)	3898 (8)	4972 (5)	3455 (5)	70 (5)	46 (4)	52 (4)	-30 (4)	11 (4)	12 (3)
O(4)	2619 (7)	3444 (5)	3212 (4)	49 (4)	42 (4)	29 (3)	-21 (3)	8 (3)	4 (3)
O(5)	-2109 (9)	3672 (6)	4804 (7)	62 (5)	40 (5)	161 (9)	20 (4)	64 (6)	18 (5)
O(6)	-3 (7)	3378 (4)	4123 (4)	35 (3)	24 (3)	46 (4)	3 (3)	10 (3)	9 (3)
O(7)	202 (7)	1791 (5)	2686 (4)	46 (4)	42 (4)	26 (3)	-10 (3)	-4 (3)	-4 (3)
O(8)	1067 (10)	-27 (8)	2180 (8)	64 (6)	93 (7)	147 (9)	8 (5)	-15 (6)	-85 (7)
O(9)	7154 (9)	2044 (8)	2421 (7)	52 (5)	99 (7)	120 (8)	1 (5)	-18 (5)	-16 (6)
O(10)	5830 (9)	4203 (6)	2129 (5)	66 (5)	67 (5)	68 (5)	-7 (4)	21 (4)	6 (4)
N(1)	594 (7)	1463 (5)	4926 (5)	26 (3)	18 (3)	28 (4)	-3 (3)	4 (3)	-1 (3)
N(2)	3173 (7)	2869 (5)	5040 (5)	30 (4)	27 (4)	26 (4)	-8 (3)	4 (3)	1 (3)
C(1)	314 (10)	338 (6)	4614 (6)	36 (5)	20 (4)	29 (4)	-7 (4)	8 (4)	0 (4)
C(2)	1789 (9)	1534 (7)	5843 (6)	36 (5)	32 (5)	23 (4)	-9 (4)	3 (4)	2 (4)
C(3)	2501 (10)	2641 (7)	5937 (6)	41 (5)	36 (5)	22 (4)	-14 (4)	5 (4)	-2 (4)
C(4)	4682 (10)	2290 (8)	5018 (7)	25 (5)	51 (6)	43 (5)	-8 (4)	-1 (4)	-2 (5)
C(5)	4491 (10)	1373 (8)	4304 (7)	31 (5)	49 (6)	53 (6)	2 (4)	16 (4)	1 (5)
C(6)	3361 (10)	4022 (7)	4862 (6)	50 (6)	30 (5)	30 (5)	-20 (4)	4(4)	-1 (4)
C(7)	3313 (10)	4180 (7)	3769 (6)	38 (5)	41 (6)	39 (5)	-15 (4)	6 (4)	6 (4)
C(8)	-911 (10)	1987 (7)	5052 (7)	29 (5)	30 (5)	49 (6)	0 (4)	15 (4)	3 (4)
C(9)	-1039 (10)	3107 (7)	4647 (7)	35 (5)	27 (5)	66 (7)	3 (4)	14 (5)	5 (5)

* For Cr thermal parameters are $\times 10^4$.

coordinate appeared to be identically 0.25, thus preventing the calculation of structure factors and phases for data with l odd. With the direct-methods program *LSAM* (Main, Woolfson & Germain, 1970) a solution for all non-hydrogen atoms was obtained.

The 1952 significant reflexions were placed on an absolute scale by Wilson's method, and converted to normalized structure amplitudes (E values) (Shiono, 1964). Several alternative sets of phases were generated from the 249 E values greater than 1.66, and an electron density map, computed from the set which produced the highest figures of merit, revealed the location of all non-hydrogen atoms with no spurious additional peaks. A structure-factor calculation based on these atomic positions and assigning to all atoms isotropic thermal parameters of 3.0 \AA^2 gave R_1 and R_2 , 0.224 and 0.227 respectively for all 1952 significant structure amplitudes

$$R_1 = \frac{||F_o| - |F_c||}{\sum |F_o|} \text{ and} \\ R_2 = \frac{[\sum w(|F_o| - |F_c|)^2 / \sum w|F_o|^2]^{1/2}}{\sum w|F_o|^2}$$

In the least-squares calculations, the function $\sum w(|F_o| - |F_c|)^2$ was minimized. After one cycle of full-matrix least-squares refinement and three of block-diagonal least-squares refinement the positional and isotropic thermal parameters converged to give $R_1 = 0.096$ and $R_2 = 0.126$. Anisotropic thermal parameters

were introduced for all the atoms and after six further block-diagonal least-squares refinement cycles all parameters had converged to give $R_1 = 0.059$ and $R_2 = 0.083$.

A difference synthesis clearly revealed all hydrogen atoms except those associated with water molecules. Inclusion of the hydrogen atoms at idealized positions (C-H distance 1.05 \AA), with isotropic thermal parameters 1 \AA^2 greater than the equivalent isotropic thermal parameter of the atom to which they are attached, significantly reduced R_1 and R_2 to 0.049 and 0.068 respectively. Block-diagonal least-squares refinement was continued with fixed hydrogen parameters and was considered complete when no parameter shift was greater than 0.1σ . The final residuals were $R_1 = 0.043$ and $R_2 = 0.058$.

A final difference synthesis had no major characteristics greater than $0.3 e \text{ \AA}^{-3}$.

The labelling scheme used is defined in Fig. 1 and the final values of the positional and vibrational parameters are listed in Table 1,* with their estimated standard deviations derived from the inverse least-squares matrix. Table 2 lists idealized hydrogen atom positions.

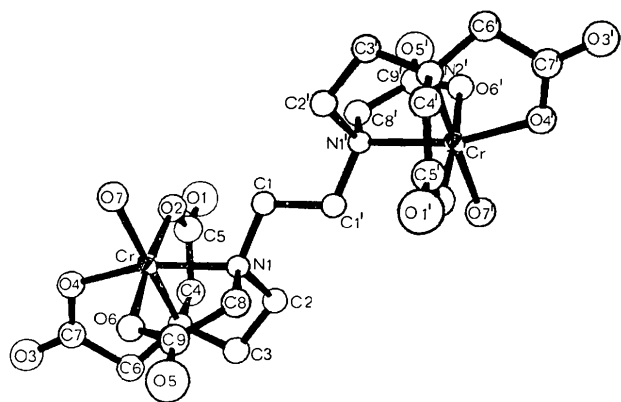


Fig. 1. A diagram of the molecule showing the labelling scheme used for the TTHA ligand.

* A list of observed and calculated structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30438 (13 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. Idealized hydrogen atom positions ($\times 10^3$)

	x/a	y/b	z/c
H(C1) <i>A</i>	139	0	449
H(C1) <i>B</i>	-49	32	395
H(C2) <i>A</i>	120	138	645
H(C2) <i>B</i>	266	97	584
H(C3) <i>A</i>	162	322	601
H(C3) <i>B</i>	338	269	656
H(C4) <i>A</i>	550	284	480
H(C4) <i>B</i>	514	202	572
H(C6) <i>A</i>	244	446	509
H(C6) <i>B</i>	443	430	524
H(C8) <i>A</i>	-187	153	469
H(C8) <i>B</i>	-99	201	580

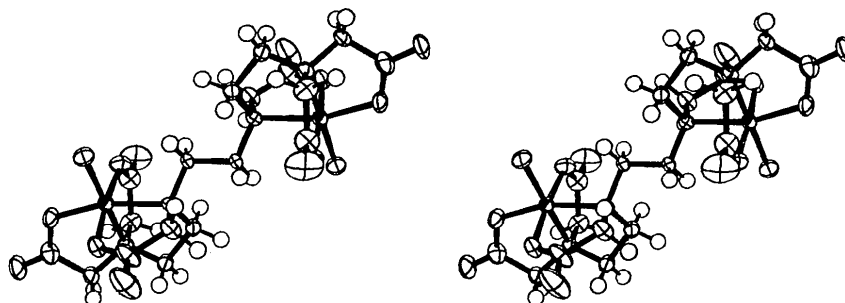


Fig. 2. A stereoscopic view of $\text{Cr}_2\text{TTHA} \cdot 2\text{H}_2\text{O}$. Thermal ellipsoids for all non-hydrogen atoms have been drawn at the 50% level, those for hydrogen have been reduced for clarity.

Scattering factors used were those tabulated by Ibers (1962) and the major programs used during refinement were modified versions of *LSAM* (Main, Woolfson & Germain, 1970), *ORFLS* (Busing, Martin & Levy, 1962), the block-diagonal least-squares program of Shiono (1968) and the Fourier summation program of White (1965). Figs. 1, 2 and 4 were drawn with the program *ORTEP* (Johnson, 1965). All major calculations were performed on the Monash University CDC 3200 computer.

Results and discussion

The structure consists of binuclear molecular complexes (a stereoscopic view is given in Fig. 2) linked by a three-dimensional array of hydrogen bonds involving the water molecules. The mid-point of the central bond connecting the pair of ethylenic carbon atoms [C(1) and C(1')] is a required centre of inversion.

The potentially decadentate TTHA ligand is utilized to its full extent in this complex, each chromium atom being octahedrally coordinated, five of the octahedral sites being occupied by atoms N(1), N(2), O(2), O(4), O(6) of the TTHA ligand and the sixth by a water molecule [O(7)]. The coordination sphere of the chromium atom forms a distorted octahedron, the lengths of the 12 edges of the octahedron varying from 2.602 to 3.095 Å as shown in Fig. 3. Moreover, the displacement of several atoms from the three least-squares planes (I, II, III, Table 5) based on the Cr atom and the six coordinating atoms are significant, the greatest distortion being apparent in plane I. The Cr atom is displaced 0.145(1) Å from the least-squares plane (IV), fitted through atoms N(1), O(2), O(4) and O(6), towards the coordinated water molecule [O(7)].

The two Cr–N bond distances differ significantly (0.08 Å, Table 3), probably as a result of differing environments of the nitrogen atoms. Bond lengths Cr–N(1), 2.120(7) Å, and Cr–N(2), 2.042(6) Å, are typical of Cr–N distances in related complexes: 2.156, 2.071 Å in *cis*-bis-(2-methoxyphenyl)-bis-(2,2'-bipyridyl)Cr(III) (Daly & Sanz, 1972), 2.068 Å (av.) in tris(glycinato)Cr-

(III) (Bryan, Greene, Stokeley & Wilson, 1971), 1.997, 2.005 Å in *N,N'*-ethylenebis(salicylideneiminato)diaquochromium(III) chloride (Coggon, McPhail, Mabbs, Richards & Thornley, 1970).

Cr–O ligand bond lengths (Table 3) may be compared with the distances 1.965(2) Å (av.) for Cr(glycinato)₃·H₂O (Bryan, Greene, Stokeley & Wilson, 1971), 1.951 Å (av.) in Cr(acac)₃ (Morosin, 1965), and 1.951(8), 1.916(8) Å [Cr–O(salen)] in CrSalenCl₂·2H₂O (Coggon *et al.*, 1970). As can be seen from Table 3 the Cr–O(4) bond length is slightly longer than either the Cr–O(2) or the Cr–O(6) bond length, probably resulting from the severe strain in the glycinato ring associated with O(4) (plane VII, Table 5). The Cr–O(7)(water) bond length of 1.997(6) Å is significantly longer (6σ) than any other Cr–O bond length in the complex, which may be caused by hydrogen bonding.

Strain within the TTHA complex can best be expressed by an analysis of the glycinato rings (Cr–O–C–N–Cr). According to Hoard and coworkers a

Table 4. Bond angles (°) with estimated standard deviations in parentheses

N(1)–Cr–O(4)	164.5 (3)		
N(2)–Cr–O(7)	173.8 (3)		
O(2)–Cr–O(6)	174.4 (3)		
C(1')–C(1)–N(1)	112.6 (6)		
N(1)–C(2)–C(3)	109.8 (6)		
N(2)–C(3)–C(2)	108.3 (6)		
Cr–N(1)–C(2)	104.6 (5)		
Cr–N(2)–C(3)	106.9 (5)		
N(1)–Cr–N(2)	85.6 (3)		
Cr–O(2)–C(5)	117.2 (6)	Cr–O(4)–C(7)	115.2 (5)
Cr–N(2)–C(4)	107.8 (5)	Cr–N(2)–C(6)	104.9 (5)
N(2)–C(4)–C(5)	112.7 (7)	N(2)–C(6)–C(7)	107.9 (7)
O(2)–C(5)–C(4)	116.5 (8)	O(4)–C(7)–C(6)	115.8 (7)
N(2)–Cr–O(2)	84.3 (3)	N(2)–Cr–O(4)	81.1 (3)
Cr–O(6)–C(9)	119.6 (6)		
Cr–N(1)–C(8)	107.4 (5)		
N(1)–C(8)–C(9)	112.3 (7)		
O(6)–C(9)–C(8)	116.2 (8)		
N(1)–Cr–O(6)	82.1 (2)		

Table 3. Bond lengths (Å) with estimated standard deviations in parentheses

Cr–N(1)	2.120 (7)	Cr–O(2)	1.946 (6)
Cr–N(2)	2.042 (6)	Cr–O(4)	1.959 (6)
Cr–O(7)	1.997 (6)	Cr–O(6)	1.943 (6)
C(1)–C(1')	1.537 (12)	N(2)–C(6)	1.488 (11)
C(1)–N(1)	1.491 (10)	C(6)–C(7)	1.522 (13)
N(1)–C(2)	1.499 (9)	C(7)–O(4)	1.290 (10)
N(2)–C(3)	1.487 (11)	C(7)–O(3)	1.231 (12)
C(2)–C(3)	1.521 (12)	N(1)–C(8)	1.494 (11)
N(2)–C(4)	1.498 (11)	C(8)–C(9)	1.516 (12)
C(4)–C(5)	1.513 (14)	C(9)–O(6)	1.290 (12)
C(5)–O(2)	1.287 (10)	C(9)–O(5)	1.214 (12)
C(5)–O(1)	1.223 (12)		

Cr–Cr distances (Å)

Intramolecular:	7.425
Intermolecular:	6.943

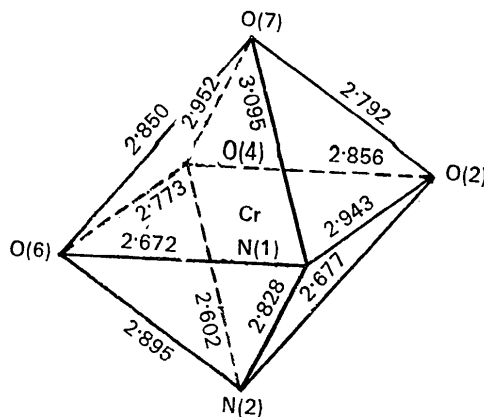


Fig. 3. Edge lengths of the coordination octahedra of chromium.

Table 5. Equations of least-squares planes and distances of individual atoms (Å) from the planes

X , Y and Z are coordinates in Å referred to an orthogonal system of axes having X along the a axis, Y in the ab plane and Z along the c^* axis. Fractional coordinates x, y, z in the system are related to X, Y, Z by the matrix equation.

$$\begin{bmatrix} 8.634 & 0 & -2.329 \\ 0 & 12.605 & 0 \\ 0 & 0 & 13.671 \end{bmatrix} \begin{bmatrix} x \\ y \\ z \end{bmatrix} = \begin{bmatrix} X \\ Y \\ Z \end{bmatrix}$$

(I) Plane through Cr, N(1), O(2), O(4), O(6)	$-0.4784X - 0.3473Y - 0.8065Z + 5.6425 = 0$			
Cr	0.116 (1)	O(2)	0.062 (6)	
		O(4)	-0.130 (6)	
N(1)	-0.126 (6)	O(6)	0.079 (6)	
(II) Plane through Cr, N(2), O(2), O(7), O(6)	$0.5015X + 0.6387Y - 0.5836Z + 0.9875 = 0$			
Cr	0.016 (1)	O(2)	-0.071 (6)	
N(2)	0.061 (6)	O(7)	0.059 (6)	
		O(6)	-0.065 (6)	
(III) Plane through Cr, N(2), N(1), O(4), O(7)	$0.6893X - 0.7140Y - 0.1226Z + 2.4711 = 0$			
Cr	0.012 (1)	N(1)	-0.109 (6)	O(7) 0.098 (6)
N(2)	0.123 (6)	O(4)	-0.124 (6)	
(IV) Plane through N(1), O(2), O(4), O(6)	$-0.4772X - 0.3472Y - 0.8073Z + 5.6750 = 0$			
N(1)	-0.100 (6)	O(4)	-0.100 (6)	Cr 0.145 (1)
O(2)	0.093 (6)	O(6)	0.106 (6)	
(V) Plane through Cr, N(1), N(2)	$0.6275X - 0.7682Y - 0.1273Z + 2.6725 = 0$			
		C(2)	0.285 (8)	C(3) -0.431 (9)
(VI) Plane through Cr, N(2), C(4), C(5), O(2)	$0.4439X + 0.6121Y - 0.6544Z + 1.5169 = 0$			
Cr	0.065 (1)	C(4)	0.071 (9)	
N(2)	-0.083 (6)	C(5)	0.002 (10)	
		O(2)	-0.054 (6)	
(VII) Plane through Cr, N(2), C(6), C(7), O(4)	$0.8638X - 0.4918Y - 0.1093Z + 1.4537 = 0$			
Cr	-0.195 (1)	C(6)	-0.237 (9)	
N(2)	0.275 (6)	C(7)	0.012 (9)	
		O(4)	0.146 (6)	
(VIII) Plane through Cr, N(1), C(8), C(9), O(6)	$-0.4595X - 0.4010Y - 0.7925Z + 5.6782 = 0$			
Cr	0.075 (1)	C(8)	0.103 (9)	
N(1)	-0.107 (6)	C(9)	-0.017 (10)	
		O(6)	-0.054 (6)	

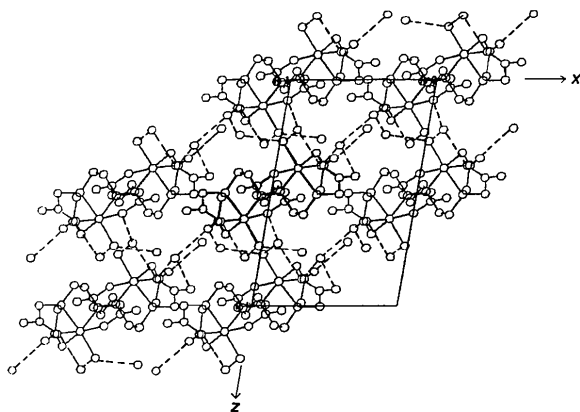


Fig. 4. Projection diagram on the ac plane depicting hydrogen bonding $A \cdots H \cdots B$ (dotted lines) in the crystal. Hydrogen atoms have been omitted for clarity.

qualitative guide to relative ring strain in the glycinate rings is provided by the deviation of the sum of the interior angles of the ring from the maximum value of 540° , with consequent loss of planarity of the ring concerned. The least-squares planes calculated for the atoms in the glycinate rings are given in Table 5 (planes VI, VII, VIII), the corresponding sum of the interior angles being 538.5 , 524.9 and 537.6° respectively (Table 4). The equatorial ring (plane VII, angle 524.9°) like those in EDTA complexes is highly strained (Lin, Leggett & Wing, 1973; Park, Glick & Hoard, 1969; Weakliem & Hoard, 1959), also having the shortest 'chelate bite' length of 2.602 Å (Fig. 3), whilst the axial rings are unstrained (planes VI, VIII).

Bond lengths within the ethylenediamine bridge, N(1)–C(2), $1.499(9)$; C(2)–C(3), $1.521(12)$; C(3)–N(2), $1.487(11)$ Å, are similar to comparable bond lengths in EDTA complexes (Stephens, 1969; Lin & Hoard, 1964; Hamor, Hamor & Hoard, 1964; Richards, Pederson, Silverton & Hoard, 1964). The conformation of the ethylenediamine bridge is expressed by the N–C–N torsion angle, 56.3° ; C(2) and C(3) being on opposite sides of the Cr, N(1), N(2) plane and respectively $0.285(8)$ and $0.431(9)$ Å from this plane.

C(1)–C(1') and C(1)–N(1) bond distances in the bridging ethylenediamine moiety [$1.537(12)$, $1.491(10)$ Å respectively] and the C(1')–C(1)–N(1) angle of $112.6(6)^\circ$ are similar to those found in bridging ethylenediamine groups in EDTA complexes: $\text{Cu}_2\text{EDTA} \cdot 4\text{H}_2\text{O}$, 1.56 , 1.52 Å, 117.5° (Fillipova *et al.*, 1973), $\text{Na}_2(\text{MoO}_3 \cdot \text{EDTA} \cdot \text{MoO}_3) \cdot 8\text{H}_2\text{O}$, 1.54 , 1.51 Å, 112° (Park, Glick & Hoard, 1969).

In solution, free rotation about the C(1)–C(1') bond would be possible and could give rise to the prediction of shorter intramolecular M–M distances than the 7.425 Å found in the solid state (Smith & Martell, 1972).

All the acetate groups were found to be planar within experimental errors.

The distances involved in hydrogen bonding are given in Table 6. Fig. 4 depicts, by broken lines, the intermolecular hydrogen-bonding scheme. Each

Table 6. Hydrogen-bond distances (Å)

	Symmetry code	Distance
O(3)—O(9)	IV	2.958
O(3)—O(10)	I	2.852
O(6)—O(8)	III	2.755
O(7)—O(8)	I	2.545
O(8)—O(2)	I	3.061
O(9)—O(10)	I	2.953
O(10)—O(1)	IV	2.891
O(9)—O(7)	II	2.615
O(10)—O(2)	IV	3.083
O(10)—O(8)	IV	2.860

Symmetry code

I	x	y	z
II	$1+x$	y	z
III	$-x$	$\frac{1}{2}+y$	$\frac{1}{2}-z$
IV	$1-x$	$\frac{1}{2}+y$	$\frac{1}{2}-z$

molecule of $\text{Cr}_2\text{TTHA} \cdot 2\text{H}_2\text{O}$ is involved in 16 hydrogen bonds with surrounding water molecules, forming an intricate three-dimensional array of hydrogen bonding. Except for hydrogen-bonded atoms, no atoms have contact distances shorter than the sum of their van der Waals contact distances.

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Etude de la Conformation Moléculaire à l'Etat Solide de la *N*-Acétyl-L-prolyl-L-lactyl-méthylamide

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(Reçu le 22 mars 1974, accepté le 23 avril 1974)

The structure of *N*-acetyl-L-prolyl-L-lactylmethylamide has been determined from three-dimensional X-ray diffractometer data. The compound crystallizes in the orthorhombic system with cell constants $a = 22.58$, $b = 8.45$, $c = 6.49$ Å, space group $P2_12_12_1$. The structure was solved by a direct method of phase determination and was refined by block-diagonal least-squares methods. The final reliability index is 0.082. The molecule shows a $\beta 1$ conformation.

Introduction

La résolution de la structure cristalline du complexe valinomycine-ion potassium (Pinkerton, Steinrauf &

Dawkins, 1969) a montré que l'enchaînement des peptides, où les fonctions ester et amide secondaire sont alternées, subit une suite de repliements stabilisés par des liaisons hydrogène du type 4→1 (IUPAC-IUB