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The Structure of the Monosodium Salt of Cytidine(5′)diphosphoethanolamine

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Abstract

$C_{11}H_{19}N_4O_{11}P_2^-.Na^+.7H_2O$, $M_r = 594{\cdot}08$, is orthorhombic, space group $P2_12_12_1$, with $a = 6{\cdot}946$ (2), $b = 12{\cdot}503$ (4), $c = 28{\cdot}264$ (8) Å, $U = 2454{\cdot}6$ Å^3, $D_x = 1{\cdot}61$ Mg m^{-3}, $Z = 4$, μ(Cu $K\alpha$) = 2·612 mm^{-1}, $F(000) = 1244$. Final $R = 0{\cdot}101$ for 1454 observed reflections. The cytosine base is in the *anti* conformation with respect to the sugar ($\chi_{CN} = 62{\cdot}6°$). The ribose exhibits an uncommon C(1′)*exo*–C(2′)*endo* puckering. The pyrophosphate has a characteristic staggered geometry. The conformation about P(2)–O(7′) is *trans* (−103·4°). This makes CDP-ethanolamine more extended compared to the folded geometry of CDP-choline, which has a *gauche* conformation (71·3°). The molecular interactions in the extended crystal structure, however, are similar to those found in CDP-choline, with the CMP-5′ portions

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tightly bound by metal ligation and the phosphoryl-ethanolamine parts only loosely held by water molecules.

Introduction

We report here the structure of cytidine(5′)diphosphoethanolamine (CDP-ethanolamine) as obtained from the crystal structure analysis of its monosodium salt. CDP derivatives are key intermediates in the metabolism of phospholipids. CDP-ethanolamine, in particular, is involved in the biosynthesis of phosphatidylethanolamine, which is an important constituent of cell membranes. The present investigation is a continuation of our earlier studies on the conformation of nucleotide coenzymes, CDP and CDP-choline (Viswamitra, Seshadri, Post & Kennard, 1975), ADP.Rb (Viswamitra, Hosur, Shakked & Kennard, 1976), ADP.K (Katti & Viswamitra, 1979), ADP free acid (Hosur & Viswamitra, 1979), UDP.K_2 (Viswamitra, Post & Kennard, 1979) and ADP-tris (Shakked, Viswamitra & Kennard, 1980). Structural studies of these coenzymes are of interest in understanding the molecular mechanisms of the group transfer reactions in which they are involved.

Experimental

Crystals of the monosodium salt of CDP-ethanolamine (from Boehringer Mannheim Biochemicals) were grown by slow diffusion of acetone into aqueous solutions of the compound. The crystals generally grew as needles. They were sealed in Lindemann-glass capillary tubes with a small quantity of the mother liquor.

The space group and cell dimensions were determined from rotation, Weissenberg and precession photographs. The cell parameters were later refined by least squares from 25 high-angle reflections collected on a CAD-4 diffractometer with crystal-monochromated Cu $K\alpha$ radiation.

Intensities to a θ limit of 60° were collected on the diffractometer from a crystal 1·95 × 0·15 × 0·025 mm, in the ω–2θ scan mode. Backgrounds for each reflection were measured at the two edges of the Bragg peak for $\frac{1}{6}$ of the scan angle. Two reflections monitored at regular intervals showed that the crystal was stable to X-rays. Of the 2070 unique reflections collected in the *hkl* octant, 1454 were retained as observed [$I > 1{\cdot}5\sigma(I)$]. These were corrected for Lorentz and polarization factors but not for absorption ($\mu r \simeq 0{\cdot}09$).

Structure solution and refinement

The structure was determined with *MULTAN* (Main, Woolfson & Germain, 1971). A fragment of 18 atoms obtained from the E map corresponding to the best set of phases (ABSFOM = 1·37, RESID = 35·21 and combined figure of merit = 1·99), was developed into the complete structure by structure-factor calculations and difference syntheses. Only three of the seven water molecules could be located. Similar difficulties in locating solvent molecules have been reported in the analyses of 5′-UMP.Na (Seshadri, Viswamitra & Kartha, 1980), 5′-UMP.Ba (Shefter & Trueblood, 1965) and 5′-IMP.Na (Rao & Sundaralingam, 1969). Block-diagonal least-squares refinement with individual isotropic temperature factors reduced R to 0·142. Further refinement with anisotropic thermal parameters converged at $R = 0{\cdot}117$. At this stage 14 of the 19 molecular H atoms were introduced at their geometrically expected positions and refined isotropically. The final $R = 0{\cdot}101$. During the final cycles the function minimized was $\sum w(|F_o| - k|F_c|)^2$, $w = 1/(a + b|F_o| + c|F_o|^2)$ with $a = 0{\cdot}89$, $b = 6{\cdot}04 \times 10^{-3}$ and $c = 5{\cdot}9 \times 10^{-4}$. Scattering factors for the non-H atoms were computed from Cromer & Waber (1965) constants and those for H were from Stewart, Davidson & Simpson (1965).

Table 1. *Final positional parameters for the non-*H *atoms* (×10⁴) *with e.s.d.'s in parentheses*

$U_{eq} = 1/(6\pi^2)\sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$.

	x	y	z	U_{eq} (Å²)
N(1)	−2066 (20)	1455 (11)	1850 (6)	31
C(2)	−2619 (26)	432 (12)	1713 (7)	32
O(2)	−4376 (16)	186 (9)	1659 (5)	39
N(3)	−1274 (18)	−356 (11)	1624 (5)	28
C(4)	607 (27)	−127 (13)	1686 (6)	30
N(4)	1856 (22)	−859 (13)	1588 (7)	48
C(5)	1200 (24)	930 (15)	1835 (8)	43
C(6)	−184 (27)	1678 (13)	1911 (8)	42
O(1′)	−3128 (17)	3105 (9)	1565 (4)	34
C(1′)	−3494 (24)	2297 (14)	1913 (8)	39
C(2′)	−3494 (24)	2829 (13)	2373 (6)	25
O(2′)	−4336 (18)	2234 (10)	2741 (4)	43
C(3′)	−4572 (24)	3868 (14)	2258 (6)	30
O(3′)	−6644 (16)	3673 (10)	2338 (5)	39
C(4′)	−4119 (23)	4042 (12)	1735 (7)	30
C(5′)	−2949 (26)	4997 (15)	1611 (7)	35
O(5′)	−1144 (15)	4949 (9)	1874 (5)	35
P(1)	437 (6)	5851 (3)	1776 (2)	21
O(11)	−372 (17)	6958 (9)	1794 (5)	37
O(12)	2123 (16)	5568 (8)	2086 (5)	33
O(6′)	962 (16)	5572 (9)	1239 (4)	31
P(2)	785 (7)	6307 (4)	770 (2)	26
O(21)	−1306 (19)	6485 (11)	648 (5)	43
O(22)	2100 (19)	7234 (10)	800 (5)	51
O(7′)	1491 (20)	5476 (11)	376 (5)	46
C(7)	3338 (33)	5474 (21)	155 (7)	58
C(8)	3141 (33)	5953 (22)	−309 (8)	63
N(5)	1615 (24)	5411 (13)	−596 (6)	45
Na	2928 (9)	3006 (5)	3122 (2)	29
O*W*(1)	4153 (18)	2589 (10)	3852 (4)	42
O*W*(2)	2958 (19)	9033 (12)	271 (6)	57
O*W*(3)	1735 (54)	6750 (28)	4412 (11)	199

The positional parameters of the heavy atoms are given in Table 1, those of H atoms in Table 2, bond lengths and angles in Tables 3 and 4, and torsion angles in Table 5.* Fig. 1 shows the chemical structure and numbering scheme.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35885 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Final positional and thermal parameters for the* H *atoms* ($\times 10^3$)

	x	y	z	U (Å^2)
H(4)	204	−168	139	34
H′(4)	306	−85	155	105
H(5)	262	114	200	100
H(6)	26	259	213	55
H(1′)	−443	203	189	69
H(2′)	−219	305	249	32
H(3′)	−428	462	247	61
H(4′)	−565	409	142	23
H(5′)	−282	492	125	22
H′(5′)	−380	562	165	47
H(7)	417	592	29	38
H′(7)	377	459	4	65
H(8)	257	681	−16	39
H′(8)	411	577	−39	50

Table 3. *Bond lengths* (Å)

N(1)–C(2)	1·390 (21)	C(4′)–C(5′)	1·487 (24)
C(2)–N(3)	1·380 (21)	C(4′)–O(1′)	1·441 (23)
C(2)–O(2)	1·267 (21)	C(5′)–O(5′)	1·459 (22)
N(3)–C(4)	1·349 (23)	O(5′)–P(1)	1·598 (12)
C(4)–N(4)	1·291 (24)	P(1)–O(6′)	1·599 (12)
C(4)–C(5)	1·447 (26)	P(1)–O(11)	1·494 (12)
C(5)–C(6)	1·358 (25)	P(1)–O(12)	1·504 (13)
C(6)–N(1)	1·348 (23)	O(6′)–P(2)	1·620 (12)
N(1)–C(1′)	1·458 (22)	P(2)–O(22)	1·478 (14)
C(1′)–C(2′)	1·459 (27)	P(2)–O(7′)	1·599 (14)
C(1′)–O(1′)	1·432 (23)	O(7′)–C(7)	1·427 (26)
C(2′)–C(3′)	1·534 (24)	C(7)–C(8)	1·447 (32)
C(2′)–O(2′)	1·406 (20)	C(8)–N(5)	1·497 (29)
C(3′)–O(3′)	1·476 (20)	P(2)–O(21)	1·509 (14)
C(3′)–C(4′)	1·527 (26)		

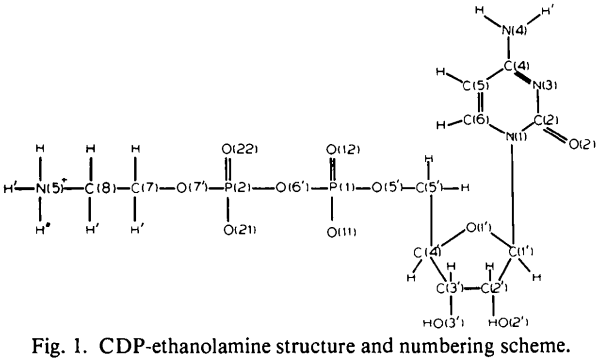


Fig. 1. CDP-ethanolamine structure and numbering scheme.

Table 4. *Bond angles* (°)

C(1′)–N(1)–C(2)	120·7 (14)	C(3′)–C(4′)–O(1′)	107·8 (13)
C(1′)–N(1)–C(6)	119·6 (15)	C(3′)–C(4′)–C(5′)	117·2 (14)
C(2)–N(1)–C(6)	119·6 (15)	O(1′)–C(4′)–C(5′)	108·2 (14)
N(1)–C(2)–N(3)	121·3 (15)	C(4′)–C(5′)–O(5′)	108·4 (14)
N(1)–C(2)–O(2)	121·5 (16)	C(5′)–O(5′)–P(1)	118·2 (11)
O(2)–C(2)–N(3)	117·2 (15)	O(5′)–P(1)–O(11)	112·9 (7)
C(2)–N(3)–C(4)	118·8 (14)	O(5′)–P(1)–O(12)	105·6 (7)
N(3)–C(4)–N(4)	118·2 (16)	O(5′)–P(1)–O(6′)	99·6 (6)
N(3)–C(4)–C(5)	120·4 (16)	O(11)–P(1)–O(12)	119·4 (7)
N(4)–C(4)–C(5)	121·2 (17)	O(11)–P(1)–O(6′)	108·6 (7)
C(4)–C(5)–C(6)	118·2 (18)	O(21)–P(1)–O(6′)	108·8 (6)
C(5)–C(6)–N(1)	121·6 (18)	P(1)–O(6′)–P(2)	129·6 (8)
N(1)–C(1′)–O(1′)	107·7 (14)	O(6′)–P(2)–O(21)	110·0 (7)
N(1)–C(1′)–C(2′)	116·0 (15)	O(6′)–P(2)–O(22)	110·6 (7)
O(1′)–C(1′)–C(2′)	106·8 (14)	O(6′)–P(2)–O(7′)	100·3 (7)
C(1′)–O(1′)–C(4′)	105·0 (13)	O(21)–P(2)–O(22)	119·5 (8)
C(1′)–C(2′)–O(2′)	114·7 (14)	O(21)–P(2)–O(7′)	103·5 (7)
C(1′)–C(2′)–C(3′)	101·4 (14)	O(22)–P(2)–O(7′)	111·1 (8)
O(2′)–C(2′)–C(3′)	113·6 (13)	P(2)–O(7′)–C(7)	125·6 (13)
C(2′)–C(3′)–O(3′)	107·7 (13)	O(7′)–C(7)–C(8)	108·1 (19)
C(2′)–C(3′)–C(4′)	103·0 (13)	C(7)–C(8)–N(5)	111·7 (19)
O(3′)–C(3′)–C(4′)	111·8 (13)		

Table 5. *Torsion angles* (°) *involving non-*H *atoms*

Average e.s.d. is 1·9°.

C(2)–N(1)–C(1′)–O(1′)	−115·5	C(4′)–C(5′)–O(5′)–P(1)	176·0
C(2)–N(1)–C(1′)–C(2′)	124·9	C(5′)–O(5′)–P(1)–O(6′)	−65·6
C(6)–N(1)–C(1′)–O(1′)	62·6	C(5′)–O(5′)–P(1)–O(11)	49·4
C(6)–N(1)–C(1′)–C(2′)	−57·0	C(5′)–O(5′)–P(1)–O(12)	−178·4
N(1)–C(1′)–C(2′)–O(2′)	−76·4	O(5′)–P(1)–O(6′)–P(2)	120·3
O(1′)–C(1′)–C(2′)–C(3′)	40·6	O(11)–P(1)–O(6′)–P(2)	2·0
C(1′)–C(2′)–C(3′)–C(4′)	−28·3	O(12)–P(1)–O(6′)–P(2)	−129·4
C(2′)–C(3′)–C(4′)–O(1′)	7·7	P(1)–O(6′)–P(2)–O(21)	−69·4
C(3′)–C(4′)–O(1′)–C(1′)	16·7	P(1)–O(6′)–P(2)–O(7′)	−178·0
C(4′)–O(1′)–C(1′)–C(2′)	−36·6	P(1)–O(6′)–P(2)–O(22)	64·7
O(1′)–C(4′)–C(5′)–O(5′)	−65·6	O(6′)–P(2)–O(7′)–C(7)	−103·4
C(3′)–C(4′)–C(5′)–O(5′)	56·4	P(2)–O(7′)–C(7)–C(8)	−101·3
O(3′)–C(3′)–C(4′)–C(5′)	130·1	O(7′)–C(7)–C(8)–N(5)	−54·5

Discussion

Cytosine base

The cytosine base is in the *anti* conformation with respect to the sugar moiety (Fig. 4*a*). The glycosidic torsion angle (χ_{CN} = 62·6°) is close to that observed in CDP-choline (60·3°). The cytosine ring is essentially planar with the exocyclic N(4) showing the maximum deviation (0·056 Å) from the best plane (Table 6). The bond lengths and angles (Tables 3, 4) correspond to those expected for the neutral cytosine base (Viswamitra, Reddy, Lin & Sundaralingam, 1971).

Ribose

The phase angle of pseudorotation is 135·3° and hence the sugar pucker is C(1′)*exo*–C(2′)*endo* (Fig. 2). This type of puckering for ribose is not commonly observed. In ADP free acid (Hosur & Viswamitra, 1979), one of the conformers of the disordered sugar assumes the C(2′)*endo*–C(1′)*exo* conformation. The mean-plane calculations for the ribose show deviations of 0·397 (17) and −0·199 (16) Å for C(1′) and C(2′)

Table 6. *Least-squares planes and deviations of atoms* (Å)

Average e.s.d. in the atom deviations is 0·017 Å.

(I) Cytosine

N(1)	0·005*	C(5)	−0·003*
C(2)	−0·012*	C(6)	−0·001*
N(3)	0·005*	O(2)	−0·028
C(4)	−0·001*	N(4)	0·056

(II) Ribose

	A	*B*
O(1′)	−0·097*	0·000*
C(1′)	0·277*	0·397
C(2′)	−0·215*	−0·199
C(3′)	0·099*	0·000*
C(4′)	0·039*	0·000*
C(5′)	−1·040	−1·119

Equations of planes

(I)	$0{\cdot}0608x + 0{\cdot}2843y - 0{\cdot}9568z = 4{\cdot}5771$
(II-*A*)	$-0{\cdot}9180x - 0{\cdot}3386y - 0{\cdot}2053z = 0{\cdot}1332$
(II-*B*)	$-0{\cdot}8762x - 0{\cdot}4135y - 0{\cdot}2477z = 0{\cdot}7972$

* Atoms used to calculate mean plane.

respectively, the latter being towards C(5′) (Table 6). The conformation about C(4′)–C(5′) is the usual *gauche–gauche* ($\varphi_{OO} = -65{\cdot}6$, $\varphi_{OC} = 56{\cdot}4°$).

Pyrophosphate

The P–O bonds to O(11), O(12) and O(21), O(22) in the two phosphate groups are nearly equal suggesting that the monoanionic charge on each phosphate is distributed between the unesterified O atoms. The two bonds from the bridging O(6′) to P(1) and P(2) are not equal [P(1)–O(6′) = 1·599, P(2)–O(6′) = 1·620 Å]. The angle P(1)–O(6′)–P(2) is 129·6°. In organic pyrophosphates, this angle is found to vary in the range 127–138°, clustering around 130°. When viewed down the P(1)–P(2) vector, the pyrophosphate group shows a staggered geometry. Its orientation in the molecule is similar to that in CDP-choline and is significantly different from that in CDP free acid, Fig. 3. The two orientations correspond to right and left rotations about C(5′)–O(5′). The torsion angle C(4′)–C(5′)–O(5′)–P(1) is 176·0° in CDP-ethanolamine, 174·2 in CDP-choline and −135·1° in CDP free acid.

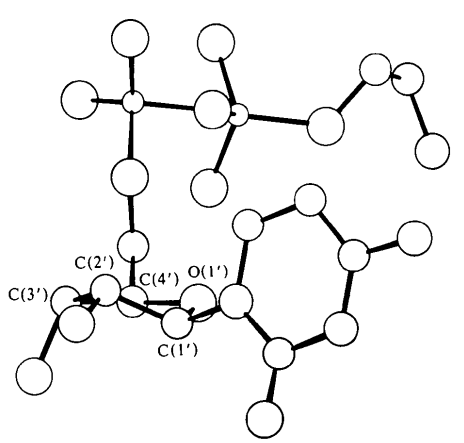
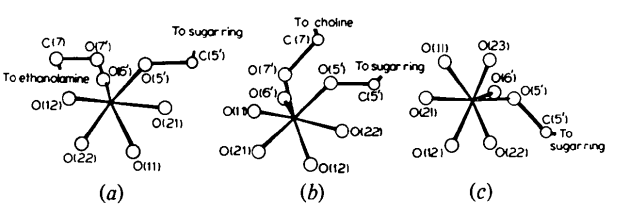


Fig. 2. View of the molecule along the C(3′)–C(4′)–O(1′) plane, showing the C(1′)*exo*–C(2′)*endo* conformation.

Ethanolamine group

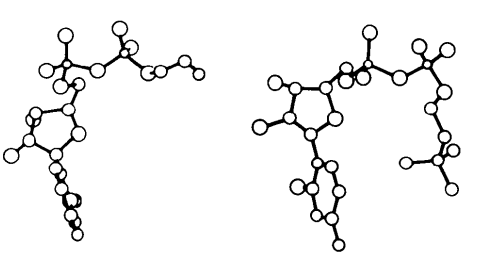
The O–C–C–N^+ group exhibits a characteristic *gauche* conformation about the C–C bond (Fig. 2). In CDP-ethanolamine, this conformation is g^- (−54·5°) in contrast to the g^+ of CDP-choline (68·8°). Both these enantiomeric *gauche* conformations have been observed in L-α-glyceryl phosphoryl choline, which has two crystallographically independent molecules (Abrahamsson & Pascher, 1966). The intramolecular non-bonded $N^+(5)\cdots O(7')$ distance in the N^+–C–C–O group is 2·75 (2) Å. It is considerably smaller than the 3·11 Å found in CDP-choline. The absence of methyl groups on $N^+(5)$ makes possible a closer contact between the N and the phosphate ester O atom in CDP-ethanolamine. In ethanolamine phosphate, the corresponding $N^+\cdots O$ distance is 2·88 Å (Kraut, 1961).

The cytidine diphosphate parts of CDP-ethanolamine and CDP-choline have similar conformational features. The most significant difference between the two molecules arises because of the rotation about P(2)–O(7′). The *trans* conformation about this bond in CDP-ethanolamine [O(6′)–P(2)–O(7′)–C(7) = −103·4°] makes it slightly extended compared to CDP-choline which is highly folded (71·3°) (Fig. 4).



(*a*) (*b*) (*c*)

Fig. 3. View down the P(1)–P(2) vector. (*a*) CDP-ethanolamine, (*b*) CDP-choline, (*c*) CDP free acid.



(*a*) (*b*)

Fig. 4. Views of (*a*) CDP-ethanolamine and (*b*) CDP-choline almost perpendicular to the sugar.

Molecular interactions and packing

Interactions of the base with the environment are mediated through O(2), N(3) and N(4). N(4) forms hydrogen bonds with the phosphate O(11) and base O(2) (Table 7). N(3) and O(2) bind to the Na^+ ion. A similar pattern of interactions has been observed in CDP-choline. In CDP free acid, which does not have any metal ions, interactions of the cytosine base are different. These are schematically shown in Fig. 5. The ribose hydroxyl O atoms take part in metal ligation as well as in intermolecular hydrogen bonding with phosphate O atoms [O(2′)···O(12) = 2·635, O(3′)···O(12) = 2·618 Å]. The Na^+ ion coordination is similar to that in CDP-choline. Five of the ligands N(3), O(11), O(2′), O(3′) and O*W*(1) are at distances ranging from 2·22 to 2·45 Å. The carbonyl O(2) is at 2·97 Å. N(5) of the ethanolamine group forms hydrogen bonds with all three water molecules as shown in Fig. 6, which is a view down **a**.

The molecules related by a *b* cell translation are linked through Na^+ ions. These are linked with 2_1 screw-related molecules by both metal ligation and hydrogen bonds. The crystal structure is so built that the CMP-5′ portions of the CDP-ethanolamine molecules are tightly bound by metal ions while the phosphorylethanolamine parts are only loosely held by water molecules. A similar mode of interaction is found in CDP-choline and it could be significant to the group transfer functions of these coenzymes.

Table 7. *Sodium and water coordination and hydrogen bonding* (Å)

Average e.s.d. in Na···O(N) = 0·015 and O···O(N) = 0·020 Å.

		Symmetry code	
Na···O(2′)	2·389	1	1 0 0
Na···O(3′)	2·387	1	1 0 0
Na···O*W*(1)	2·293	1	0 0 0
Na···N(3)	2·455	2	0 0 0
Na···O(11)	2·220	2	0 −1 0
Na···O(2)	2·970	2	0 0 0
N(4)···O(2)	2·932	1	1 0 0
N(4)···O(11)	3·192	1	0 −1 0
O(12)···O(3′)	2·618	1	1 0 0
O(2′)···O(12)	2·635	2	0 −1 0
O(21)···O*W*(1)	2·794	2	0 0 0
O(22)···O*W*(1)	2·817	2	1 0 0
N(5)···O*W*(1)	2·994	4	0 1 −1
O(22)···O*W*(2)	2·765	1	0 0 0
O(21)···O*W*(2)	2·727	3	−1 1 0
N(5)···O*W*(2)	2·788	3	−1 1 0
N(5)···O*W*(3)	2·936	4	0 1 −1

Symmetry code: (1) x,y,z; (2) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (3) $\frac{1}{2}+x, \frac{1}{2}-y, -z$; (4) $\frac{1}{2}-x, -y, \frac{1}{2}+z$. The numbers following the symmetry code indicate cell translations of the atom.

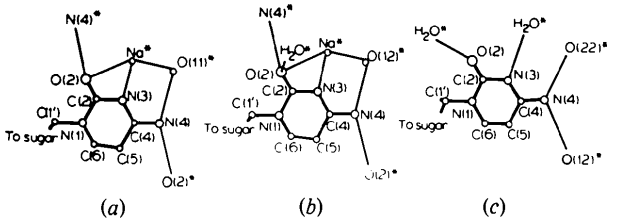


Fig. 5. Intermolecular interactions involving the base in (*a*) CDP-ethanolamine, (*b*) CDP-choline, (*c*) CDP free acid. An asterisk marks atoms related by crystallographic symmetry.

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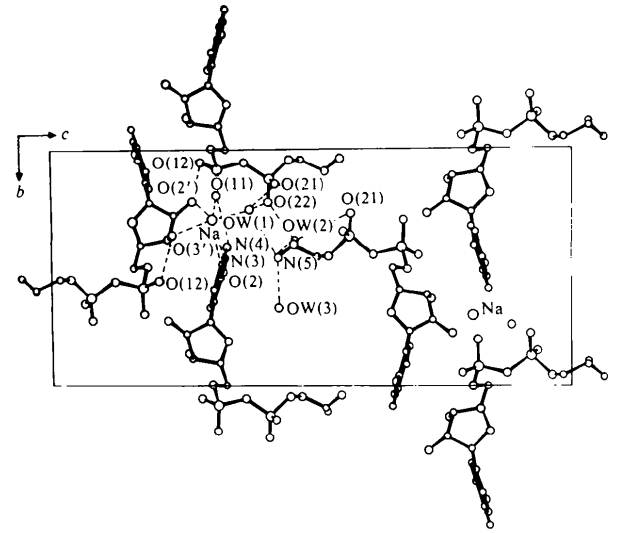

Fig. 6. Projection of the extended crystal structure on the *bc* plane.

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Structure of Di-μ-hydroxo-bis{bis[(*S*)-alaninato]chromium(III)} Trihydrate

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Abstract

The title compound, $C_{12}H_{26}Cr_2N_4O_{10}\cdot 3H_2O$, belongs to the triclinic space group $P\bar{1}$, with $a = 5{\cdot}369$ (4), $b = 9{\cdot}994$ (3), $c = 11{\cdot}209$ (2) Å, $\alpha = 72{\cdot}65$ (2), $\beta = 74{\cdot}76$ (3), $\gamma = 77{\cdot}27$ (3)°, $Z = 1$, $D_c = 1{\cdot}65$, $D_o = 1{\cdot}64$ (1) Mg m^{-3}. The structure was refined on 2073 independent nonzero reflections to a conventional R factor of 0·031. The crystal consists of individual dihydroxo-bridged molecules in which each Cr atom is surrounded by a *cis–cis–cis* arrangement of two hydroxo O atoms, and two O and two N atoms of alanine. The molecules are held together by an intricate network of hydrogen bonds involving amino groups, free and coordinated O atoms of carbonyl groups and water molecules.

Introduction

Chromium(III) forms two common types of complexes with simple bidentate amino acids (HL): monomeric CrL_3 chelates and dihydroxo-bridged dimers of the formula $Cr_2(OH)_2L_4$. The tris-chelates can exist either as *facial* or as *meridional* isomers. The glycine complex studied by Bryan, Greene, Stokely & Wilson (1971) was found to be the *fac* isomer. For the dihydroxo-bridged molecules, 24 stereoisomers are theoretically possible, *i.e.* 10 pairs of enantiomers and 4 inactive diastereoisomers. The glycine compound examined by Veal, Hatfield, Jeter, Hempel & Hodgson (1973) had a *cis–cis–cis* distribution of donor atoms in the $CrN_2O_2O'_2$ coordination sphere. For an amino acid with an asymmetric C atom, like (*S*)-alanine (HAla), the 24 stereoisomers above are all diastereoisomers. As part of an investigation of Cr complexes with amino acids, we have prepared crystals of $Cr_2(OH)_2(S\text{-Ala})_4$ and a crystallographic study was undertaken in order to determine the stereochemistry around Cr.

(*S*)-Alanine (0·4 g) and $[Cr(NH_3)_6](NO_3)_3$ (0·5 g) were dissolved in water (15 ml). While the mixture was heated on a steam bath (1 h), pink needles of $Cr(S\text{-Ala})_3$ formed. After 3 d at room temperature, a small amount of burgundy crystals of $Cr_2(OH)_2(S\text{-Ala})_4$ had also appeared. The solid was filtered and the burgundy crystals were separated by hand. A similar compound had been prepared by Oki & Otsuka (1976) from racemic alanine, but Oki (1977) failed to obtain the analog with (*S*)-alanine.

A well formed crystal of dimensions 0·35 × 0·15 × 0·11 mm was used for X-ray work. Triclinic Laue symmetry was observed on a set of precession and cone-axis photographs. A cell-reduction calculation using program *TRACER* II (Lawton & Jacobson, 1965) showed that the lattice cannot be described with a cell of higher symmetry. The preparation of the compound from (*S*)-alanine unambiguously ruled out the centrosymmetric $P\bar{1}$ group, leaving $P1$ as the only acceptable space group.

The crystal was mounted on an Enraf–Nonius CAD-4 diffractometer. The cell parameters were obtained by least-squares refinement on the setting angles of 25 reflections ($12° < 2\theta < 24°$). The cell used in this report is non-conventional. It can be transformed into a conventional Niggli reduced cell of type II (*International Tables for X-ray Crystallography*, 1969) using $\mathbf{a}_r = -\mathbf{a}$, $\mathbf{b}_r = \mathbf{b}$, $\mathbf{c}_r = \mathbf{a} - \mathbf{c}$. The parameters of the reduced cell are: $a_r = 5{\cdot}369$, $b_r =$

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