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Structure of Disodium Deoxycytidine 5′-Phosphate Heptahydrate, $C_9H_{12}N_3O_7P^{2-}.2Na^+.7H_2O$

By J. Pandit, T. P. Seshadri and M. A. Viswamitra

Department of Physics and ICMR Centre on Genetics and Cell Biology, Indian Institute of Science, Bangalore 560 012, India

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Abstract. $M_r = 477·3$, orthorhombic, $P2_12_12_1$, $a = 6·719(4)$, $b = 29·614$ (15), $c = 9·559$ (3) Å, $Z = 4$, $U = 1902·0$ Å^3, $D_x = 1·67$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 1·90$ mm^{-1}, $T = 290$ K. Final R for 1809 observed reflections is 0·045. The structure shows an unusual *gauche–trans* conformation about the C(4′)—C(5′) bond, while the sugar pucker [C(3′)-*exo*] and glycosidic torsion angle [$\chi_{CN} = 70·2$ (5)°, *anti*] are normal. The two Na$^+$ ions do not interact with the molecule directly, being completely surrounded by water molecules. The cytosine bases are stacked, with a separation distance of 3·36 (5) Å.

Introduction. We have for some time been investigating the crystal structures of DNA constituents. We had earlier reported the structure of deoxycytidine 5′-phosphate (dCMP) as a part of these studies (Viswamitra, Reddy, Lin & Sundaralingam, 1971). We have now obtained the structure of its disodium salt (5′-dCMPNa$_2$.7H$_2$O). The present structure is significantly different from that of the free acid, showing an unusual *gauche–trans* conformation about the C(4′)—C(5′) bond, accompanied, however, by a normal [C(3′)-*exo*] sugar pucker.

Experimental. Crystals grown from aqueous solutions of the compound by diffusion of acetone, crystal data obtained from rotation and Weissenberg photographs and refined by least-squares calculations from 25 strong reflections, CAD-4 diffractometer, Cu $K\alpha$ intensity data up to $\sin\theta/\lambda = 0·60$ Å^{-1} collected on the diffractometer in ω–2θ scan mode, 1809 out of 1945 reflections considered observed [$F > 3\sigma(F)$], background counts measured for $\frac{1}{6}$ total scan angle on either side of Bragg peak, two strong reflections monitored periodically during data collection showed crystal stable to X-rays; structure solved by a combination of Patterson (Sheldrick, 1976) and direct methods (Main, Woolfson & Germain, 1971), the two Na$^+$ ions and seven water O atoms located from successive difference Fourier maps; block-diagonal least-squares refinement with anisotropic thermal parameters reduced R to 0·063; a difference map computed at this stage revealed all H atoms present in structure, except for one belonging to a water molecule; further refinement including H atoms and weights $w = 1/\sigma^2(F_o)$ converged at $R = 0·045$, $R_w = 0·064$; $F(000) = 960$, scattering factors for non-H atoms computed from function developed by Cromer & Waber (1965), for H atoms from Stewart, Davidson & Simpson (1965).

Discussion. The molecular structure and atomic numbering are shown in Fig. 1. The final atomic parameters are listed in Table 1.* Interatomic distances and angles are given in Table 2.

The conformation of the cytosine base with respect to the furanose ring is *anti*. The glycosidic torsion angle χ[C(6)—N(1)—C(1′)—O(4′) = 70·2 (5)°] is in significant contrast to the value of −5·9° reported for dCMP free acid.

The bond angle at N(3) [C(2)—N(3)—C(4)] is 119·5 (4)°, normally expected for the unprotonated cytosine ring. Considerable widening of this angle has been observed in protonated bases such as dCMP free acid, where the corresponding angle is 123·9 (2)°.

The only exocyclic atom to show a significant deviation from the plane of the base is the carbonyl O(2). It is displaced by 0·157 (4) Å, approximately in the direction of its hydrogen bonds.

C(3′) of the deoxyribose moiety deviates by 0·465 (5) Å on the opposite side of C(5′) from the least-squares plane defined by C(4′)—O(4′)—C(1′) — C(2′). The furanose ring conformation can therefore be described as C(3′)-*exo*, which is the same as that of dCMP free acid. The pseudorotation parameters calculated from the ring torsion angles* are: phase angle of pseudorotation (P) = 186·0 (1)° and maximum amplitude of pucker (τ_m) = 31·2 (5)°.

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* Lists of structure factors, anisotropic temperature factors, least-squares planes, torsion angles involving the ribose ring, stacking interactions <3·5 Å, sodium coordination angles and Fig. 6 (Na$^+$ coordination polyhedra) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38189 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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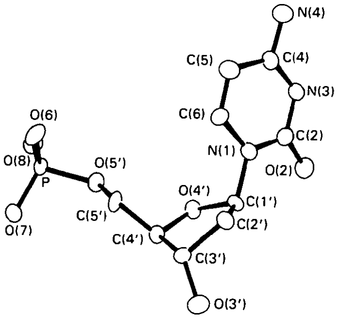

Fig. 1. Molecular structure and atomic numbering.

Table 1. *Final positional parameters* (×10^4; *for* H ×10^3) *and isotropic temperature factors with e.s.d.'s in parentheses*

$$B_{\rm eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}/B_{\rm iso}$(Å^2)
N(1)	4063 (6)	2435 (1)	2219 (4)	1·75 (8)
C(2)	4239 (7)	2881 (2)	1810 (5)	1·93 (11)
N(3)	4163 (6)	2990 (1)	442 (4)	2·09 (10)
C(4)	4094 (7)	2663 (2)	−513 (5)	1·99 (11)
C(5)	4110 (8)	2198 (2)	−145 (5)	2·24 (11)
C(6)	4056 (8)	2099 (2)	1231 (5)	2·03 (12)
O(2)	4464 (5)	3178 (1)	2714 (5)	2·39 (8)
N(4)	4031 (7)	2779 (2)	−1877 (4)	2·65 (11)
C(1′)	4084 (7)	2311 (2)	3726 (5)	1·60 (10)
C(2′)	2569 (8)	1957 (2)	4115 (6)	1·98 (11)
C(3′)	3630 (7)	1671 (2)	5199 (5)	1·78 (10)
C(4′)	5803 (7)	1701 (2)	4750 (5)	1·77 (10)
O(4′)	6003 (5)	2124 (1)	4041 (4)	1·89 (7)
O(3′)	3487 (6)	1845 (1)	6600 (4)	2·98 (10)
C(5′)	6326 (7)	1306 (2)	3811 (5)	1·88 (11)
O(5′)	8363 (5)	1351 (1)	3363 (4)	2·04 (8)
O(6)	8102 (5)	634 (1)	1974 (4)	2·43 (8)
O(7)	9666 (6)	622 (1)	4362 (4)	2·59 (8)
O(8)	11430 (5)	1021 (1)	2345 (4)	2·49 (8)
P	9479 (2)	877·5 (4)	2981 (1)	1·50 (2)
Na(1)	10787 (3)	361 (1)	8240 (2)	2·54 (5)
Na(2)	14877 (3)	917 (1)	8107 (2)	3·04 (5)
O*W*(1)	8266 (6)	875 (1)	6985 (4)	2·95 (9)
O*W*(2)	10976 (6)	−249 (1)	4254 (4)	2·78 (9)
O*W*(3)	9000 (6)	0 (1)	10086 (4)	3·12 (10)
O*W*(4)	13021 (6)	586 (1)	6202 (4)	2·68 (9)
O*W*(5)	1601 (6)	3778 (1)	10046 (4)	3·02 (9)
O*W*(6)	−724 (5)	4647 (1)	7874 (4)	2·66 (8)
O*W*(7)	6691 (6)	3900 (1)	10607 (4)	2·90 (10)
HC(6)	416 (11)	176 (2)	164 (8)	5·8 (15)
HC(5)	427 (16)	195 (3)	−75 (10)	6·2 (23)
H1N(4)	392 (14)	259 (2)	−259 (8)	6·7 (22)
H2N(4)	383 (15)	311 (3)	−206 (10)	10·1 (27)
HC(1′)	405 (12)	263 (2)	423 (7)	5·2 (15)
H1C(2′)	225 (9)	177 (2)	319 (6)	4·2 (11)
H2C(2′)	135 (9)	209 (2)	447 (6)	5·1 (11)
HC(3′)	299 (8)	137 (2)	530 (6)	3·1 (10)
HO(3′)	229 (14)	186 (2)	686 (9)	5·8 (20)
HC(4′)	646 (11)	171 (2)	568 (7)	3·8 (13)
H1C(5′)	619 (7)	102 (1)	439 (5)	3·5 (8)
H2C(5′)	550 (10)	128 (2)	302 (7)	4·4 (14)
H1*W*(1)	848 (9)	81 (2)	599 (6)	5·4 (11)
H2*W*(1)	884 (9)	115 (2)	718 (6)	4·6 (11)
H1*W*(2)	1075 (11)	−34 (2)	512 (8)	7·9 (17)
H2*W*(2)	1030 (11)	1 (2)	409 (9)	6·5 (19)
H1*W*(3)	846 (11)	14 (2)	1071 (7)	8·2 (15)
H1*W*(4)	1385 (12)	33 (2)	591 (8)	7·5 (17)
H2*W*(4)	1196 (15)	66 (3)	551 (10)	12·1 (28)
H1*W*(5)	237 (11)	357 (2)	1009 (7)	5·4 (14)
H2*W*(5)	220 (13)	400 (2)	931 (8)	8·1 (18)
H1*W*(6)	68 (9)	453 (2)	784 (6)	4·7 (11)
H2*W*(6)	−161 (10)	435 (2)	771 (7)	7·2 (14)
H1*W*(7)	592 (11)	362 (2)	1070 (8)	7·5 (17)
H2*W*(7)	642 (14)	402 (3)	978 (9)	9·1 (22)

The torsion angles $\varphi_{\rm OO}$[O(5′)—C(5′)—C(4′)—O(4′)] and $\varphi_{\rm OC}$[O(5′)—C(5′)—C(4′)—C(3′)] are 60·2 and 178·0°. The nucleotide therefore shows the *gauche–trans* (*gt*) conformation, in significant contrast to the *gauche–gauche* (*gg*) conformation found in dCMP free acid. The difference between these two conformations, as seen approximately parallel to the plane of the sugar, is shown in Fig. 2. The interesting feature of this conformation is that it is accompanied by a normal [C(3′)-*exo*] sugar pucker. Other metal complexes of nucleotides which have an unusual conformation about C(4′)—C(5′) are always accompanied by a rare sugar pucker (Swaminathan & Sundaralingam, 1979). The $\mathrm{dC}_p\mathrm{G}_p\mathrm{C}_p\mathrm{G}_p\mathrm{C}_p\mathrm{G}$ hexanucleotide (Wang, Quigley, Kolpak, Crawford, van Boom, van der Marel & Rich, 1979) also exhibits a *gt* conformation. However, it has a *syn* conformation for the dG residues. A *gt* instead of the *gg* conformation has also been incorporated at 'kinks' in the DNA backbone in the model for the highly folded structure of DNA in chromatin proposed by Crick & Klug (1975).

The phosphate group is a double anion, with two negative charges distributed between the three O atoms. This is evident from the fact that the P—O bond distances are all nearly equal (Table 2).

Table 2. *Bond distances* (Å) *and angles* (°) *not involving* H *atoms with e.s.d.'s in parentheses*

N(1)—C(2)	1·383 (6)	C(3′)—C(4′)	1·524 (7)
C(2)—N(3)	1·348 (6)	C(4′)—O(4′)	1·432 (6)
N(3)—C(4)	1·332 (6)	O(4′)—C(1′)	1·434 (6)
C(4)—C(5)	1·420 (7)	C(3′)—O(3′)	1·439 (6)
C(5)—C(6)	1·348 (7)	C(4′)—C(5′)	1·516 (7)
C(2)—O(2)	1·242 (6)	C(5′)—O(5′)	1·440 (6)
C(4)—N(4)	1·349 (6)	P—O(5′)	1·632 (4)
N(1)—C(1′)	1·487 (6)	P—O(6)	1·517 (4)
C(1′)—C(2′)	1·507 (7)	P—O(7)	1·527 (4)
C(2′)—C(3′)	1·517 (7)	P—O(8)	1·505 (4)

C(2)—N(1)—C(6)	119·9 (4)	C(2′)—C(3′)—C(4′)	103·0 (4)
C(2)—N(1)—C(1′)	120·6 (4)	C(2′)—C(3′)—O(3′)	113·8 (4)
C(6)—N(1)—C(1′)	119·2 (4)	C(4′)—C(3′)—O(3′)	107·7 (4)
N(1)—C(2)—N(3)	119·9 (4)	C(3′)—C(4′)—O(4′)	105·9 (4)
N(1)—C(2)—O(2)	119·4 (4)	C(3′)—C(4′)—C(5′)	110·1 (4)
N(3)—C(2)—O(2)	120·7 (4)	O(4′)—C(4′)—C(5′)	111·9 (4)
C(2)—N(3)—C(4)	119·5 (4)	C(1′)—O(4′)—C(4′)	110·6 (3)
N(3)—C(4)—C(5)	122·3 (4)	C(4′)—C(5′)—O(5′)	108·9 (4)
N(3)—C(4)—N(4)	118·5 (4)	C(5′)—O(5′)—P	115·0 (3)
C(5)—C(4)—N(4)	119·1 (4)	O(5′)—P—O(6)	105·7 (2)
C(4)—C(5)—C(6)	116·9 (5)	O(5′)—P—O(7)	105·7 (2)
N(1)—C(6)—C(5)	120·9 (4)	O(5′)—P—O(8)	104·4 (2)
N(1)—C(1′)—C(2′)	113·8 (4)	O(6)—P—O(7)	111·3 (2)
N(1)—C(1′)—O(4′)	107·9 (3)	O(6)—P—O(8)	114·1 (2)
C(2′)—C(1′)—O(4′)	106·7 (4)	O(7)—P—O(8)	114·6 (2)
C(1′)—C(2′)—C(3′)	103·8 (4)		

Sodium coordination distances (Å); average e.s.d. = 0·004 Å

Na(1)—O*W*($6^{\rm i}$)	2·374	Na(2)—O*W*($5^{\rm v}$)	2·308
—O*W*($3^{\rm ii}$)	2·392	—O*W*($2^{\rm iii}$)	2·346
—O*W*($2^{\rm iii}$)	2·406	—O*W*($4^{\rm ii}$)	2·423
—O*W*($7^{\rm iv}$)	2·533	—O*W*($1^{\rm vi}$)	2·522
—O*W*($4^{\rm ii}$)	2·560	—O*W*($7^{\rm iv}$)	2·542
—O*W*($1^{\rm ii}$)	2·579		

Symmetry code: (i) $1-x,-\frac{1}{2}+y,\frac{3}{2}-z$; (ii) x,y,z; (iii) $\frac{5}{2}-x,-y,\frac{1}{2}+z$; (iv) $\frac{1}{2}+x,\frac{1}{2}-y,2-z$; (v) $\frac{3}{2}+x,\frac{1}{2}-y,2-z$; (vi) $1+x,y,z$.

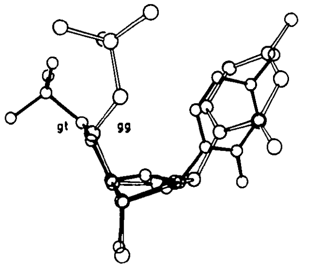

Fig. 2. Superposition of 5′-dCMPNa$_2$ (dark bonds) on 5′-dCMP free acid (open bonds) viewed approximately along the plane of the sugar ring.

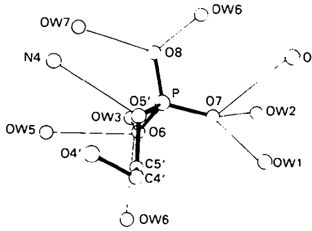

Fig. 3. Environment of the phosphate group. Thin lines indicate hydrogen bonds.

The hydrogen bonding around the phosphate group is shown in Fig. 3. The phosphate O atoms are completely surrounded by water molecules, and they interact with the positively charged Na$^+$ ions only through the water O atoms.

Theoretical studies by Berthod & Pullman (1977) show the preference of through-water binding of Na$^+$ ions to a phosphate anion compared with direct interaction. The intermediate molecules of water can be considered to serve as waters of hydration both of the anion and of the cation.

Comparison with other Na$^+$ salts of mononucleotides shows that provided the extent of hydration is large, the Na$^+$ ions interact with the phosphate only indirectly, through water bridges. Na$^+$ does not bind directly to the phosphate O atoms in the crystal structures of 5′-GMPNa$_2$.7H$_2$O (Katti, Seshadri & Viswamitra, 1981), 5′-dAMPNa.6H$_2$O (Reddy & Viswamitra, 1975) and 5′-IMPNa.6H$_2$O (Rao & Sundaralingam, 1969). However, when the extent of hydration is not so large, the phosphate O atoms also provide a ligand for sodium coordination, as has been observed in the crystal structures of 5′-dGMPNa$_2$.4H$_2$O (Viswamitra & Seshadri, 1974; Young, Tollin & Wilson, 1974) and 5′-dUMPNa$_2$.5H$_2$O (Viswamitra, Seshadri & Post, 1980).

The planar pyrimidine rings show a considerable degree of overlap as seen in Figs. 4 and 5. The separation distance is 3·36 (5) Å and stacking is propagated from one unit cell to the next by the *a*-axis repeat. The distance between the sugar O(4′) and N(4) of the adjacent base is 2·915 (7) Å which is nearly equal to the sum of the van der Waals radii of the two atoms. There is, however, no hydrogen bonding between the two atoms. A list of all other stacking interactions [minimum 3·293 (7) Å] has been deposited.*

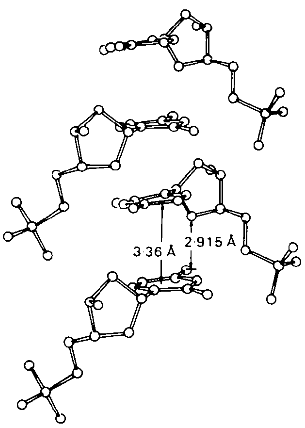

Fig. 4. Base stacking viewed down **c**. The base–base as well as the sugar–base stacking interactions are indicated.

Table 3. *Hydrogen-bond distances* (Å) *and angles* (°)

Average e.s.d.'s in distances not involving H atoms = 0·007 Å, in distances involving H = 0·075 Å, in angles = 0·4°.

A–H···*B*	*A*···*B*	*A*–H	*A*–H···*B*	H···*B*	Unit-cell translation of atom *B*		
O*W*(3)–H1*W*(3)···O(6^{i})	2·673	0·804	153·0	1·923	0	0	1
O*W*(6)–H1*W*(6)···O(6^{iii})	2·706	1·007	171·7	1·706	−1	0	1
O*W*(5)–H2*W*(5)···O(6^{iii})	2·789	1·042	176·8	1·747	−1	0	1
O*W*(1)–H1*W*(1)···O(7^{i})	2·781	0·980	160·9	1·836	0	0	0
O*W*(4)- H2*W*(4)···O(7^{i})	2·861	0·995	163·3	1·893	0	0	0
O*W*(2)–H2*W*(2)···O(7^{i})	2·727	0·916	155·3	1·868	0	0	0
O*W*(6)–H2*W*(6)···O(8^{iii})	2·758	1·063	163·3	1·723	−2	0	1
O*W*(7)–H2*W*(7)···O(8^{iii})	2·837	0·892	149·4	2·033	−1	0	1
O(3′)–HO(3′)···O(2^{iii})	2·782	0·841	172·6	1·946	−1	0	1
O*W*(1)–H2*W*(1)···O(2^{iii})	2·932	0·934	164·3	2·021	0	0	1
O*W*(4)–H1*W*(4)···O*W*(3^{ii})	2·857	0·985	159·8	1·912	2	0	−1
O*W*(2)–H1*W*(2)···O*W*(6^{iv})	2·768	0·881	160·9	1·920	1	−1	1
O*W*(5)–H1*W*(5)···N(3^{i})	2·924	0·794	172·1	2·135	0	0	1
N(4)–H2N(4)···O(5′^{iii})	2·976	1·004	152·5	2·049	−1	0	0

Symmetry code: (i) x,y,z; (ii) $\frac{1}{2}-x,\bar{y},\frac{1}{2}+z$; (iii) $\frac{1}{2}+x,\frac{1}{2}-y,\bar{z}$; (iv) $\bar{x},\frac{1}{2}+y,\frac{1}{2}-z$.

Hydrogen-bond distances and angles are listed in Table 3. The phosphate ester O(5′) is involved in a hydrogen bond with N(4) of the adjacent base. This is likely to be among the factors that contribute to the significant increase in length of the P—O(5′) bond observed in the present structure [1·632 (4) Å compared to 1·597 (2) Å in dCMP free acid].

Of the three phosphate O atoms, O(6) and O(7) form three hydrogen bonds, while O(8) forms two hydrogen bonds, all with water O atoms.

The hydroxyl O(3′) of the sugar forms a nearly linear hydrogen bond with the carbonyl O(2) of an adjacent base.

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* See deposition footnote.

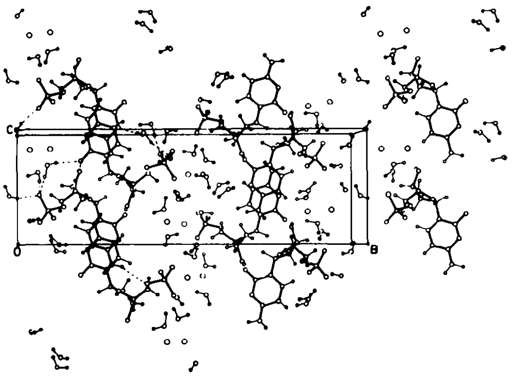

Fig. 5. The molecular packing viewed down **a**. Hydrogen bonds are indicated by broken lines.

The two cations are completely surrounded by water molecules (Fig. 6).* Na(1) has six neighbours in a nearly octahedral environment. Na(2) is pentacoordinated, sharing four of the water molecules with Na(1). The coordination polyhedra extend in infinite chains along **a**. Coordination distances are given in Table 2, angles have been deposited.*

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* See deposition footnote.

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Bis[μ-ammoniomethyl(methyl)phosphinato-*O*,*O′*]-diaquatetrachlorodicopper(II), $Cu_2(C_2H_8NO_2P)_2Cl_4(H_2O)_2$

By W. Sawka-Dobrowolska and T. Głowiak

Institute of Chemistry, University of Wrocław, 14 *Joliot-Curie,* 50–583 *Wrocław, Poland*

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Abstract. $C_4H_{20}Cl_4Cu_2N_2O_6P_2$, $M_r = 523{\cdot}05$ triclinic, $P\bar{1}$, $a = 8{\cdot}975\ (2)$, $b = 8{\cdot}658\ (2)$, $c = 6{\cdot}326\ (2)$ Å, $\alpha = 90{\cdot}32\ (1)$, $\beta = 117{\cdot}34\ (1)$, $\gamma = 94{\cdot}37\ (1)^\circ$, $V = 434{\cdot}9$ Å^3, $Z = 1$, $D_m = 1{\cdot}99\ (1)$ (by flotation), $D_x = 2{\cdot}00$ Mg m^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 10{\cdot}65$ mm^{-1}, $F(000) = 262$, $R = 0{\cdot}058$ for 1139 observed counter reflexions. The coordination sphere of the Cu atom is square pyramidal, consisting of two O atoms from different phosphinato groups, two Cl atoms and one water molecule. The Cu atoms are bridged by two phosphinato groups, giving rise to an eight-membered ring. The Cu—Cu distance is 5·081 (2) Å. The ligand exists as a zwitterion.

Introduction. The structure of the title compound has been determined as one of a series of complexes of aminomethylphosphonic and aminomethyl(methyl)phosphinic acids (AMPh and AMMPh, respectively) with divalent metal ions. So far only the crystal structures of the six compounds [Mn(AMMPh)$_2$(H$_2$O)$_2$]Br$_2$.2H$_2$O (Głowiak & Sawka-Dobrowolska, 1977*a*), [MnCl$_2$(AMMPh)$_2$(H$_2$O)$_2$] (Głowiak & Sawka-Dobrowolska, 1977*b*), [Cu(AMPh)$_2$] (Głowiak, Sawka-Dobrowolska, Jeżowska-Trzebiatowska & Antonów, 1980*a*), [Zn(AMMPh)$_2$Cl$_2$] (Žák, Kožíšek & Głowiak, 1981), [Zn(AMPh)$_2$].4H$_2$O (Fenot, Darriet & Garrigou-Lagrange, 1978), [Co(AMPh)$_2$(H$_2$O)$_2$].H$_2$O (Głowiak, Sawka-Dobrowolska, Jeżowska-Trzebiatowska & Antonów, 1980*b*) have been investigated. A typical feature of these complexes is their polymeric nature. Of the six complexes investigated, only one is monomeric. In this group of compounds the

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