order to define the general conformation of the complex we will focus on the six-membered ring formed by atoms Rh(1), P(1), C(51), Fe(1), C(61) and P(2); we do not intend to imply that this ring is of significance in the usual chemical sense. The conformation of this central fragment of the complex is shown in Fig. 2. An analysis of the ring by the method of Cremer & Pople (1975) shows that the ring has a large puckering amplitude ($Q = 0.567$), with angular deviations $\theta_2 = 234.9$, $\varphi_2 = 123.8°$. This corresponds to a half-boat conformation with the atom P(1) furthest from the mean plane of the ring. Bond angle Rh—P(2)—C(51) [$125.8 (1)°$] is substantially larger than Rh—P(1)—C(61) [$116.5 (1)°$].

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Comparison of Two Hydrated Forms of Sodium Inosine 5'-Monophosphate

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Abstract. The crystal and molecular structure of a decahydrated form of the sodium salt of inosine 5'-monophosphate ($C_{10}H_{12}N_4O_8P^-.Na^+.10H_2O$) was solved to study the effect of hydration on the conformation of nucleic acids. Monoclinic, space group $P2_1$, $a = 8.730 (3)$, $b = 22.349 (7)$, $c = 12.282 (4)$ Å, $\beta = 109.68 (3)°$, $V = 2256.52$ Å^3, $M_r = 550.34$, $Z = 4$, $F(000) = 1196$, $D_x = 1.62$ g cm^{-3}, $\mu = 21.7$ cm^{-1}, $\lambda(Cu\ K\alpha) = 1.54056$ Å, $R = 0.070$, $wR = 0.102$ for 3404 unique [$I_{net} > 2\sigma(I_{net})$] observed reflections out of 3457 unique reflections. The two molecules (A and B) in the asymmetric unit differ in the arrangement of the first shell of hydration and in the torsion angles of the ribose and phosphate. The bond lengths and angles are similar to those of the structure of a less hydrated ('dry') form of the same nucleotide ($C_{10}H_{12}N_4O_8P^-.Na^+.8H_2O$) determined previously in space group $C222_1$ [Rao & Sundaralingam (1969). *J. Am. Chem. Soc.* **91**, 1210–1217]. The twofold symmetry in the 'dry' form is destroyed in the present crystal structure due to the relative displacement of the two independent molecules and reorganization of the hydration shell. Molecule A is different from (r.m.s. $= 0.190$ Å) while molecule B is similar to (r.m.s. $= 0.093$ Å) that of the 'dry' form. The conformation adopted is influenced mainly by the differences in the endocyclic torsion angles of the ribose.

Introduction. The importance of the hydration shell in determining the conformation of the nucleic acids, and hence their function, has been widely recognized (Saenger, 1984). The crystal structure of sodium inosine 5'-monophosphate (5'-IMP; $C_{10}N_4O_8H_{12}P^-.Na^+.8H_2O$), $R = 0.10$, has been reported (Rao & Sundaralingam, 1969). In this 'dry' form structure, only six of the water molecules

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Table 1. *Fractional atomic coordinates* $(\times 10^4)$ *and equivalent isotropic thermal parameters* B_{iso} $(\text{Å}^2 \times 10)$ *for 5'-IMP*

	x	y	z	B_{iso}
Na1	00901 (04)	02554 (02)	09060 (02)	047 (01)
O3P*A*	−03915 (05)	02548 (02)	01633 (04)	034 (02)
P*A*	−02602 (02)	02969 (00)	02331 (01)	028 (01)
O1P*A*	−01055 (05)	02925 (02)	02027 (04)	035 (02)
O2P*A*	−03222 (05)	03606 (02)	02282 (04)	034 (02)
O5'*A*	−02053 (05)	02748 (02)	03653 (04)	033 (02)
C5'*A*	−03272 (08)	02777 (03)	04184 (05)	034 (03)
O4'*A*	−02447 (05)	01827 (02)	05160 (04)	036 (02)
C4'*A*	−02758 (07)	02458 (03)	05322 (05)	029 (02)
O3'*A*	−01396 (05)	02610 (02)	07377 (04)	038 (02)
C3'*A*	−01222 (07)	02679 (03)	06276 (05)	028 (02)
O2'*A*	01431 (05)	02184 (02)	07249 (04)	038 (02)
C2'*A*	00063 (07)	02242 (03)	06221 (05)	029 (02)
C1'*A*	−00930 (07)	01663 (03)	05988 (06)	031 (03)
N1*A*	01850 (06)	−00346 (02)	06784 (05)	034 (02)
C2*A*	01413 (09)	00019 (03)	07485 (06)	041 (03)
N3*A*	00678 (07)	00545 (03)	07191 (05)	038 (03)
C4*A*	00455 (07)	00666 (03)	06073 (05)	030 (03)
C5*A*	00894 (07)	00344 (03)	05280 (06)	032 (03)
C6*A*	01637 (08)	−00240 (03)	05615 (06)	035 (03)
O6*A*	02081 (07)	−00601 (03)	05058 (05)	053 (03)
N7*A*	00440 (07)	00631 (03)	04226 (05)	039 (03)
C8*A*	−00265 (09)	01124 (03)	04378 (06)	037 (03)
N9*A*	−00263 (06)	01185 (02)	05498 (05)	032 (02)
Na2	02067 (04)	03838 (02)	10654 (02)	049 (01)
O3P*B*	04154 (05)	03830 (02)	18072 (04)	036 (02)
P*B*	04782 (02)	03395 (01)	17374 (01)	027 (01)
O1P*B*	06609 (04)	03440 (02)	17650 (04)	035 (02)
O2P*B*	04164 (05)	02762 (02)	17450 (04)	034 (02)
O5'*B*	04039 (05)	03590 (02)	16034 (03)	032 (02)
C5'*B*	02298 (07)	03525 (03)	15507 (05)	035 (03)
O4'*B*	02029 (05)	04478 (02)	14530 (04)	031 (02)
C4'*B*	01718 (07)	03847 (03)	14368 (05)	029 (02)
O3'*B*	01293 (05)	03729 (02)	12338 (04)	038 (02)
C3'*B*	02442 (07)	03646 (03)	13454 (05)	030 (03)
O2'*B*	04218 (05)	04205 (02)	12602 (04)	037 (02)
C2'*B*	03770 (07)	04106 (03)	13606 (05)	028 (02)
C1'*B*	02945 (07)	04673 (03)	13830 (05)	027 (02)
N1*B*	05318 (07)	06701 (03)	13501 (05)	039 (03)
C2*B*	04295 (10)	06351 (04)	12671 (06)	044 (03)
N3*B*	03789 (08)	05817 (03)	12852 (05)	041 (03)
C4*B*	04406 (07)	05661 (03)	13992 (05)	028 (02)
C5*B*	05439 (07)	05978 (03)	14890 (06)	033 (03)
C6*B*	05950 (07)	06554 (03)	14676 (06)	037 (03)
O6*B*	06832 (07)	06928 (02)	15338 (05)	054 (03)
N7*B*	05716 (07)	05667 (03)	15896 (05)	041 (03)
C8*B*	04893 (09)	05168 (03)	15596 (06)	041 (03)
N9*B*	04058 (06)	05137 (02)	14459 (04)	031 (02)
*W*1	09732 (04)	00140 (02)	02168 (03)	026 (02)
*W*2	01422 (06)	03544 (03)	08603 (04)	044 (02)
*W*3	02808 (06)	02781 (03)	10958 (04)	046 (02)
*W*4	08699 (06)	04253 (03)	07250 (05)	053 (03)
*W*5	04663 (07)	04074 (03)	10315 (05)	051 (03)
*W*6	00268 (08)	01378 (03)	10769 (05)	058 (03)
*W*7	05694 (09)	02165 (03)	09482 (06)	071 (04)
*W*8	08686 (06)	07082 (03)	07612 (05)	054 (03)
*W*9	00613 (09)	04713 (04)	09821 (06)	074 (04)
*W*10	02384 (11)	04814 (04)	07241 (10)	099 (06)
*W*11	04348 (11)	06393 (04)	07917 (08)	089 (05)
*W*12	05431 (13)	00287 (04)	09303 (11)	116 (07)
*W*13	11871 (08)	00728 (05)	01922 (06)	082 (04)
*W*14	02104 (08)	05901 (05)	08854 (07)	082 (05)
*W*15	02484 (20)	01548 (07)	09516 (15)	182 (13)
*W*16	09516 (22)	00771 (05)	09047 (10)	159 (11)
*W*17	08785 (13)	03531 (10)	09880 (07)	221 (16)
*W*18	−01018 (09)	02251 (07)	10001 (08)	111 (06)
*W*19	06565 (23)	04868 (09)	09116 (17)	207 (16)
*W*20	06770 (13)	05835 (06)	09414 (10)	118 (07)

B_{iso} is the sum of the diagonal elements of the orthogonalized U_{ij} coefficients matrix times $\frac{8}{3}\pi^2$.

Table 2. *Torsion angles* (°) *of inosine 5'-mono-phosphate in two hydrated crystal forms*

		IMP.10H₂O*		IMP.6H₂O†
		Molecule *A*	Molecule *B*	
O1P—P—O5'—C5'		176·7 (4)	173·6 (4)	176
O2P—P—O5'—C5'		56·7 (3)	52·5 (3)	55
O3P—P—O5'—C5'	(α)	−63·8 (3)	−66·6 (3)	−65
P—O5'—C5'—C4'	(β)	169·3 (6)	167·0 (6)	172
O5'—C5'—C4'—C3'	(γ)	60·3 (4)	59·3 (4)	59
C5'—C4'—C3'—O3'	(δ)	145·7 (6)	149·1 (7)	148
O4'—C1'—N9'—C4	(χ)	−135·7 (6)	−138·3 (6)	−135
C4'—O4'—C1'—C2'	(ν_0)	−29·0 (3)	−22·4 (3)	−21
O4'—C1'—C2'—C3'	(ν_1)	42·1 (3)	38·3 (3)	37
C1'—C2'—C3'—C4'	(ν_2)	−38·6 (3)	−38·5 (3)	−39
C2'—C3'—C4'—O4'	(ν_3)	22·8 (3)	26·4 (3)	27
C3'—C4'—O4'—C1'	(ν_4)	4·0 (3)	−2·6 (3)	−4
Pseudorotation phase angle		156·4	165·1	167·4
τ_m		31·7	23·2	21·5
Sugar pucker		C2'-*endo*	C2'-*endo*	C2'-*endo*

* This work.
† Rao & Sundaralingam (1969).

occupying nine sites could be accounted for per 5'-IMP. As part of a study for the effect of H_2O and ^2H_2O on the conformation of nucleotides, we here report the structure of a decahydrated, 'wet' form of 5'-IMP and discuss the impact of the shell of hydration on the conformation.

Experimental. The crystals of 5'-IMP formed from a solution containing 20 mg ml^{-1} of 5'-IMP, and 10% 2-methyl-2,4-pentanediol, equilibrated against 40% 2-methyl-2,4-pentanediol by vapor diffusion at room temperature. The crystals were mounted in capillaries for data collection, as they deteriorated quickly upon exposure to air. The complete three-dimensional intensity data were recorded from two crystals as the crystals abruptly lost quality after a period of data collection, perhaps due to reordering of the water molecules in the crystal lattice.

Two rectangular crystals, both with dimensions $0.2 \times 0.2 \times 0.4$ mm, were used for data collection on a Rigaku AFC-5*R* rotating anode diffractometer, using graphite-monochromated Cu *K*α radiation, with ω-scan method. $(\sin\theta)/\lambda < 0.56$ Å^{-1}, lattice parameters are calculated from 15 reflections with $10 < 2\theta < 25°$. $2\theta_{max} = 120.9°$ and $-9 \leq h \leq 9$, $0 \leq k \leq 25$, $0 \leq l \leq 13$. No absorption correction was applied. Three reflections were monitored as intensity standards with total decay < 2%. Reflections measured from these two crystals were merged $(R_{int} = 3.71\%)$. A total of 3457 unique reflections were used to obtain a set of 3404 $I_{net} > 2\sigma(I_{net})$ unique reflections which were used in refinement. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). The first *E* map revealed 22 non-H atoms of the molecule. Several cycles of refinement followed by difference synthesis, using *NRCVAX* package (Gabe, Lee & Le Page, 1985), revealed all the remaining non-H atoms. Atom P*A* was fixed along the *y* axis, to define the origin. H atoms were located at this stage from the difference map. In the final cycle of refinement, there were 91 atoms with 613 parameters, $R = 0.070$, $wR = 0.102$, goodness-of-fit, $\{\sum w[(kF_o)^2 - (F_c)^2]^2/(NREF-NVAR)\}^{1/2} = 4.876$, based on F_o, $w = 1/[\sigma^2(F_o) + (0.02F_o)^2]$. Located and calculated H atoms were refined in the final stages of refinement. In the final cycle $(\Delta/\sigma)_{max} = 0.040$. The

final $\Delta\rho$ map has $(\Delta\rho)_{max} = 0.66$ and $(\Delta\rho)_{min} = -0.62$ e Å^{-3}. Atomic scattering factors calculated by the analytical form using the coefficients in *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 72–75).

Discussion. The final atomic parameters [atom and torsion angle names as per standard nomenclature for nucleic acids (Saenger, 1984)] and the comparative torsion angles are given in Tables 1* and 2 respectively. The two crystal forms are closely related [$a \sim a'$, $b \sim b'$ and $c\cos(\beta - 90°) \sim 0.5\ c'$; prime for 'dry' form]. The present structure is somewhat better refined. The bond distances and angles are in good agreement between the two crystal forms.

In the present 'wet' form structure, the volume occupied by each hydrated 5'-IMP is 564·1 Å^3, which is slightly larger than that of the 'dry' form structure (552·2 Å^3). The difference, ~12 Å^3, is less than a single water molecule would occupy in liquid water. We found ten fully occupied distinct water molecules in the 'wet form' structure, in contrast to the nine partially occupied sites in the 'dry' structure. This new water organization produces a different crystal structure in which the two independent 5'-IMP molecules adopt slightly different conformations (r.m.s. = 0·20 Å). Fig. 1(a) compares the two 5'-IMP's of the asymmetric unit. Fig. 1(b) compares the 5'-IMP pairs in the 'wet' and the 'dry' forms, with a view down a pseudo-twofold screw axis, drawn by *ORTEP*II (Johnson, 1976). The molecule A (wet) is shifted relative to that in the 'dry' form.

Fig. 2. examines the molecules surrounding the pseudo-twofold axis, along with the first shell of hydration and two Na ions. The exact twofold symmetry is broken by the water arrangement about the two molecules A and B (in black bonds). For example, the two Na ions have different coordinations. Na1 has sixfold coordination, while Na2 has sevenfold coordination, with the seventh ligand being water W17 (2·78 Å). The average coordination distances around Na1 and Na2 are 2·45 (2·32–2·78 Å) and 2·51 (2·35–2·81 Å), respectively. Na2 is relatively displaced from its corresponding O2' and has an additional weak coordination with W17.

The details for the differences in the torsion angles between the 'dry' and 'wet' are found mainly in the endocyclic torsion angles of the sugars (Table 2). The 'dry' form has endocyclic torsion angles closer to molecule B than molecule A, while the torsion about

the glycosyl bond χ is more similar between the 'dry' form and molecule A, though all three molecules are in the *anti* conformation. All ribose rings in both forms adopt the C2'-*endo* pucker conformation, which is unusual for ribose. The overall crystal packing features, with alternate layes of nucleotides and water channels in which the Na ions are embedded, is retained in the 'dry' and the 'wet' forms. The
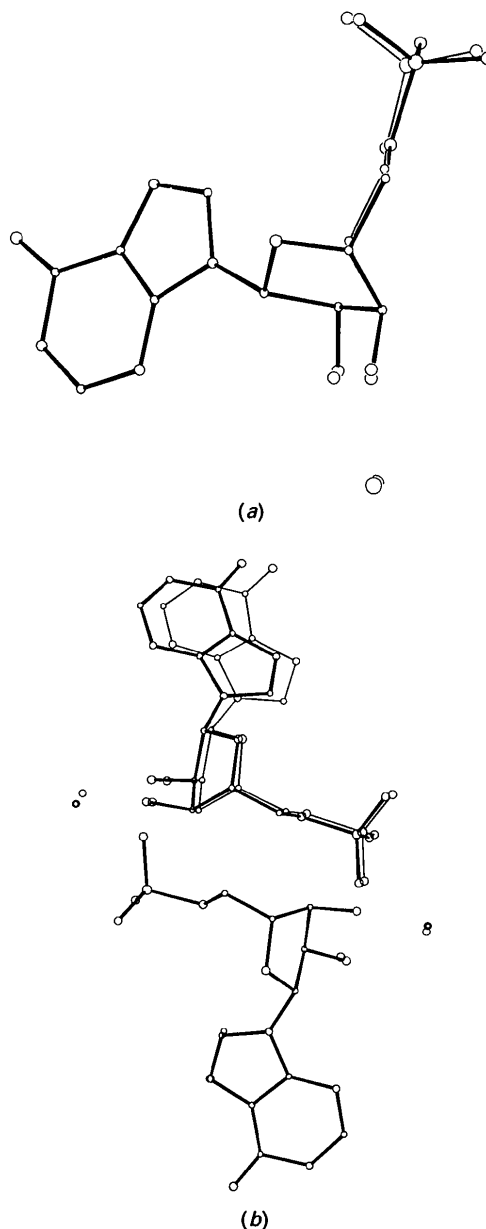

(a)

(b)

Fig. 1. (a) Least-squares superposition of the two independent 5'-IMP molecules in the decahydrated crystal form. (b) Superposition of the two independent molecules in the 'wet' form (thick lines) with the twofold related 5'-IMP molecules in the 'dry' crystal form (thin lines).

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* Lists of structure factors, anisotropic thermal parameters, bond angles and distances, hydrogen bonds and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53454 (37 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
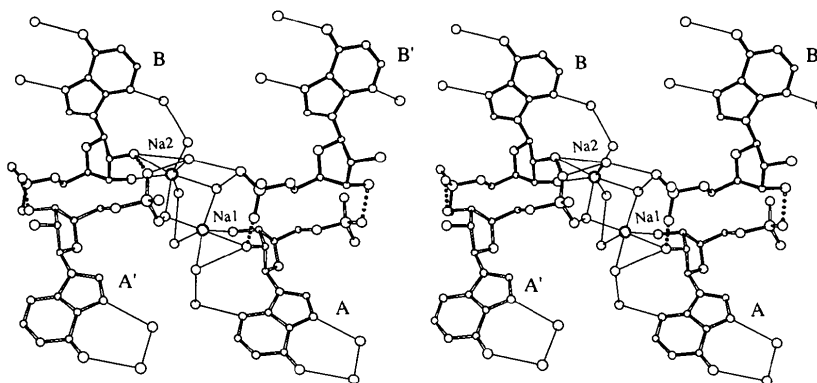
Fig. 2. Stereoscopic crystal packing diagram of the 'wet' form structure showing the alternating layers of 5'-IMP and water channel in which the two Na ions (circles with thick lines) are embedded.

two 5'-IMP molecules A and B are directly linked together using four strong hydrogen bonds (2·65–2·74 Å) between the phosphates and the ribose O2' and O3' hydroxyls, forming an infinite network (Fig. 2). In the water channels, some loosely bound water molecules could occur and cause the sharp change in the diffraction quality of the crystal either by moving in or escaping from the lattice.

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Structure of Bis(di-2-pyridylamine)copper(I) Chloride

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Abstract. $[Cu(C_{10}H_9N_3)_2]Cl$, $M_r = 441·40$, monoclinic, $P2_1/n$, $a = 9·675$ (2), $b = 16·838$ (3), $c = 12·618$ (1) Å, $\beta = 105·91$ (1)°, $V = 1976·8$ Å^3, $Z = 4$, $D_m = 1·482$, $D_x = 1·48$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0·71073$ Å, $\mu = 12·6$ cm^{-1}, $F(000) = 904$, $T = 294$ (1) K, $R = 0·031$ for 2850 observed reflections. The Cu atom is in a distorted tetrahedral environment of four N atoms from two di-2-pyridylamine ligands, with an average Cu—N distance of 2·016 (2) Å. The geometries of the di-2-pyridylamine ligands are quite different. The dihedral angle between the pyridyl rings is 5·6 (4)° for one ligand and 16·7 (2)° for the other. The chloride counterions form hydrogen-bonded bridges between adjacent complexes.

Introduction. Dipyridylamine has received interest as a ligand due to its ability to stabilize five-coordinate copper(II) complexes. The flexibility of the molecule at the amine N atom is manifested in the wide range