catechol O atoms with Br···N distances of 3·247 (5), 3·264 (6) Å for (II), 3·280 (3), 3·303 (4) Å for (III) and Br···O distances of 3·351 (5), 3·305 (4) Å for (II), 3·336 (3), 3·574 (4) Å for (III). Angles at H range from 145 (7) to 161 (4)° for (II) and 127 to 166° for (III). These interactions describe a distorted square-planar 'coordination geometry' about the bromide ion. The hydroxyl group on the *p*-hydroxyphenyl substituent O(3) acts as a donor to atom O(2) in both structures with an O···O separation of 2·820 (6) Å for (II) and 2·811 (4) Å for (III); the angle at H is 173 (7)° for (II) and 157 (6)° for (III). In both (II) and (III) the catechol H(O1) atom may participate in a three-center interaction involving the adjacent halogen. Angles about H(O1), [O(1)–H(O)···Cl = 126 (5), O(1)–H(O1)···Br = 127 (5)°] are equivalent for (III), whereas angles for (II) [O(1)–H(O1)···F = 112 (6), O(1)–H(O1)···Br = 145 (7)°] show a polarization away from F towards the Br ion.

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Structure of 1-Methyladenosine Trihydrate

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Abstract. N^6-Didehydro-1,6-dihydro-1-methyladenosine trihydrate, $C_{11}H_{15}N_5O_4.3H_2O$, $M_r = 335·3$, monoclinic, space group $P2_1$, $a = 6·726$ (1), $b = 20·090$ (3), $c = 11·496$ (11) Å, $\beta = 91·22$ (6)°, $V = 1553$ (1) Å^3, $Z = 4$, $D_m = 1·427$ (3), $D_x = 1·434$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 0·995$ mm^{-1}, $F(000) = 712$, $T = 296$ K, $R = 0·049$ for 2687 observed reflections. The molecular conformations of the two independent nucleosides in the asymmetric unit are quite different. The conformation around the glycosidic bond is *syn* in molecule (*a*), whereas it is *anti* in molecule (*b*). The conformation of the exocyclic C(4′)–C(5′) bond is *gauche-gauche* in molecule (*a*) and *trans-gauche* in molecule (*b*). On the other hand, the sugar puckerings of both molecules are the C(2′)-*endo* type. The most pronounced feature of the crystal structure is the alternating parallel stacking of the two independent purine bases to form columns parallel to the *a* axis.

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Introduction. A number of alkylated nucleosides occur in nature or are obtained by chemical modification. Among these, 1-methyladenosine is one of the minor nucleosides isolated from transfer RNA in many eukaryotes and some microorganisms (Nishimura, 1978). According to the three-dimensional structure reported for yeast $tRNA^{Phe}$ (Landner *et al.*, 1975; Quigley *et al.*, 1975; Stout *et al.*, 1978), 1-methyladenosine (m^1A58) is located at the $T\psi C$ loop and forms a reverse Hoogsteen-type base pairing with a thymine base (T54). It is also stacked on the paired bases between G18 of the *D* loop and $\psi 55$. These structural features seem to be necessary for retaining the sharp bend in the $T\psi C$ loop and for stabilizing the interaction between the $T\psi C$ and *D* loops. In addition, 1-methyladenine base can be produced when single-strand DNA and RNA are treated with alkylating agents which are mutagenic or carcinogenic. The

alkylation at the N(1) position of adenine may induce some conformational perturbations in the secondary or tertiary polynucleotide structure. It is hence imperative to obtain the precise molecular conformation of 1-methyladenosine and compare it with the related nucleosides.

Experimental. 1-Methyladenosine was synthesized as previously described (Jones & Robins, 1963) and crystallized as colorless needles from an acetone solution containing a small amount of water. The crystal density was determined by flotation in C_6H_6–CCl_4. A crystal of dimensions $0{\cdot}1 \times 0{\cdot}1 \times 0{\cdot}4$ mm was used for data collection on a Rigaku automatic four-circle diffractometer with graphite-monochromatized Cu $K\alpha$ radiation. The unit-cell dimensions were calculated by a least-squares procedure based on 2θ values ($31 < 2\theta < 54°$) of 15 reflections. The intensity fluctuation (<2%) was monitored periodically with three reflections (200, 060, 002). 2769 independent intensities ($2\theta_{max} = 135°$, $0 \le h \le 7$, $0 \le k \le 23$, $-13 \le l \le 13$) were measured with the ω–2θ scan technique and corrected for Lorentz and polarization factors. The structure was solved by the Patterson and minimum function methods and refined by the block-diagonal least-squares method with anisotropic temperature factors for all non-H atoms. All H atoms, located on a difference Fourier map, were included with constant isotropic temperature factors, The final R was 0·049 ($wR = 0{\cdot}058$, $S = 1{\cdot}48$) for 2687 reflections with $F_o \neq 0$. The function minimized was $\sum w(|F_o| - k|F_c|)^2$ where $w = [\sigma^2(F_o) - 0{\cdot}0583|F_o| + 0{\cdot}0045|F_o|^2]^{-1}$ and k is the scale factor. The highest and lowest peaks in the final difference Fourier map had heights of 0·20 and −0·29 e Å^{-3}, respectively. The average and maximum shift/e.s.d. ratios for atomic parameters were 0·07 and 0·28, respectively. All numerical calculations were carried out on an ACOS 850 computer at the Crystallographic Research Center, Institute for Protein Research, Osaka University, using the programs of *The Universal Crystallographic Computation Program System–Osaka* (1979) and their modifications. The atomic scattering factors used were taken from *International Tables for X-ray Crystallography* (1974).

Discussion. The final atomic parameters for non-H atoms are listed in Table 1.*

The bond distances and angles are shown in Table 2. For the most part, corresponding bond distances and angles for the two independent molecules (*a*) and (*b*) agree; the greatest differences are observed in the C(4)–C(5) and N(7)–C(8) bond distances and the C(2′)–C(1′)–O(4′) and C(2′)–C(3′)–O(3′) bond angles. Since methylation of N(1) produces the imino form of the adenine ring, some characteristic features of the imino form are observed in the bond distances and angles of the 1-methyladenine ring as compared to the normal adenine residue (Taylor & Kennard, 1982). The bond distances and angles in the imidazole moiety are in good agreement with standard values in the adenine residue, while in the pyrimidine moiety, the bond distances and angles, in particular around the C(6) atom, are significantly different from those of the normal adenine residue. For example, the N(1)–C(6) bond is longer by *ca* 0·06 Å than that of the normal adenine base, whereas the C(6)–N(6) bond is shorter by *ca* 0·05 Å. Also, the

Table 1. *Final positional and equivalent isotropic temperature parameters for non-H atoms with e.s.d.'s in parentheses*

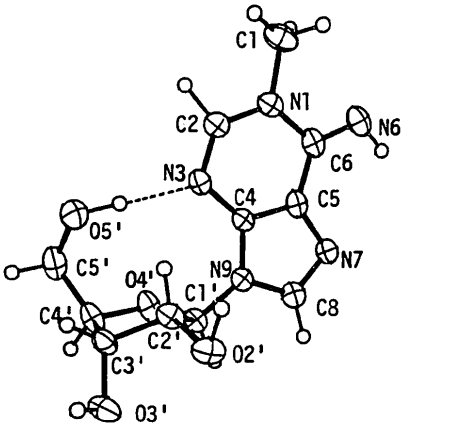
$B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}(\mathbf{a}_i.\mathbf{a}_j)$.

	x	y	z	B_{eq}(Å^2)
Molecule (*a*)				
N(1)	0·0328 (4)	−0·0272 (2)	0·1826 (3)	2·3 (1)
C(2)	0·0376 (5)	−0·0926 (2)	0·1492 (3)	2·2 (1)
N(3)	0·0328 (4)	−0·1440 (1)	0·2181 (3)	2·2 (1)
C(4)	0·0221 (5)	−0·1266 (2)	0·3318 (3)	1·9 (1)
C(5)	0·0194 (5)	−0·0624 (2)	0·3767 (3)	2·1 (1)
C(6)	0·0298 (5)	−0·0064 (2)	0·2991 (3)	2·2 (1)
N(7)	0·0060 (5)	−0·0628 (2)	0·4961 (3)	2·5 (1)
C(8)	−0·0008 (6)	−0·1270 (2)	0·5217 (3)	2·5 (1)
N(9)	0·0081 (4)	−0·1679 (2)	0·4269 (2)	2·1 (1)
C(1)	0·0342 (6)	0·0241 (2)	0·0903 (4)	3·4 (1)
N(6)	0·0345 (5)	0·0560 (2)	0·3243 (3)	3·2 (1)
C(1′)	−0·0021 (5)	−0·2400 (2)	0·4288 (3)	2·0 (1)
C(2′)	−0·1640 (5)	−0·2698 (2)	0·3493 (3)	2·3 (1)
C(3′)	−0·0832 (6)	−0·3395 (2)	0·3267 (3)	2·5 (1)
C(4′)	0·1406 (6)	−0·3274 (2)	0·3239 (3)	3·0 (1)
C(5′)	0·2227 (6)	−0·3221 (2)	0·2029 (4)	3·5 (1)
O(4′)	0·1787 (3)	−0·2652 (1)	0·3858 (2)	2·7 (1)
O(2′)	−0·3534 (4)	−0·2682 (1)	0·3998 (2)	2·8 (1)
O(3′)	−0·1308 (5)	−0·3834 (1)	0·4189 (3)	3·8 (1)
O(5′)	0·1126 (5)	−0·2765 (2)	0·1307 (2)	3·8 (1)
Molecule (*b*)				
N(1)	0·5328 (4)	−0·0095 (2)	0·3706 (2)	2·3 (1)
C(2)	0·5399 (5)	0·0242 (2)	0·2689 (3)	2·2 (1)
N(3)	0·5410 (4)	−0·0013 (1)	0·1645 (2)	2·2 (1)
C(4)	0·5347 (5)	−0·0697 (2)	0·1674 (3)	1·8 (1)
C(5)	0·5236 (5)	−0·1082 (2)	0·2640 (3)	1·7 (1)
C(6)	0·5216 (5)	−0·0796 (2)	0·3780 (3)	2·0 (1)
N(7)	0·5194 (4)	−0·1753 (1)	0·2340 (2)	2·0 (1)
C(8)	0·5284 (5)	−0·1753 (2)	0·1215 (3)	2·1 (1)
N(9)	0·5401 (4)	−0·1124 (1)	0·0748 (2)	1·9 (1)
C(1)	0·5310 (7)	0·0272 (2)	0·4800 (3)	3·4 (1)
N(6)	0·5131 (5)	−0·1083 (2)	0·4772 (2)	2·6 (1)
C(1′)	0·5306 (5)	−0·0950 (2)	−0·0488 (3)	1·9 (1)
C(2′)	0·6670 (5)	−0·1361 (2)	−0·1248 (3)	2·0 (1)
C(3′)	0·5491 (5)	−0·1371 (2)	−0·2407 (3)	2·0 (1)
C(4′)	0·3347 (5)	−0·1417 (2)	−0·1988 (3)	2·2 (1)
C(5′)	0·2596 (6)	−0·2115 (2)	−0·1803 (3)	3·0 (1)
O(4′)	0·3336 (3)	−0·1063 (1)	−0·0892 (2)	2·6 (1)
O(2′)	0·8627 (3)	−0·1127 (2)	−0·1283 (2)	3·1 (1)
O(3′)	0·5864 (4)	−0·0766 (1)	−0·2984 (2)	2·6 (1)
O(5′)	0·2021 (4)	−0·2418 (2)	−0·2879 (2)	3·3 (1)
Water				
O(*W*1)	−0·1277 (4)	−0·3191 (1)	0·7540 (3)	3·5 (1)
O(*W*2)	−0·5102 (6)	0·0930 (2)	0·9685 (3)	4·8 (1)
O(*W*3)	0·7724 (5)	−0·3013 (2)	0·9878 (3)	4·4 (1)
O(*W*4)	0·6118 (5)	−0·4572 (2)	0·2501 (3)	4·8 (1)
O(*W*5)	−0·5232 (4)	0·1940 (2)	0·3750 (3)	4·1 (1)
O(*W*6)	0·0200 (6)	−0·5033 (2)	0·2547 (3)	5·4 (1)

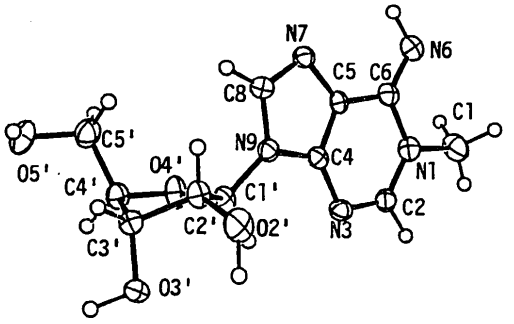
* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, hydrogen bonds and conformational parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44106 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond lengths* (Å) *and angles* (°) *for* 1-*methyladenosine trihydrate*

	Mol. (*a*)	Mol. (*b*)		Mol. (*a*)	Mol. (*b*)
N(1)–C(2)	1·370 (5)	1·352 (5)	N(9)–C(1′)	1·451 (4)	1·463 (4)
N(1)–C(6)	1·404 (5)	1·414 (5)	C(1′)–C(2′)	1·528 (5)	1·524 (5)
N(1)–C(1)	1·480 (5)	1·458 (5)	C(1′)–O(4′)	1·415 (4)	1·413 (4)
C(2)–N(3)	1·302 (5)	1·305 (5)	C(2′)–C(3′)	1·525 (5)	1·536 (5)
N(3)–C(4)	1·356 (4)	1·375 (4)	C(2′)–O(2′)	1·412 (4)	1·399 (4)
C(4)–C(5)	1·388 (5)	1·356 (4)	C(3′)–C(4′)	1·526 (5)	1·533 (5)
C(4)–N(9)	1·378 (4)	1·369 (4)	C(3′)–O(3′)	1·421 (5)	1·411 (4)
C(5)–C(6)	1·439 (5)	1·431 (5)	C(4′)–C(5′)	1·510 (6)	1·507 (5)
C(5)–N(7)	1·378 (5)	1·392 (4)	C(4′)–O(4′)	1·458 (5)	1·446 (4)
C(6)–N(6)	1·286 (5)	1·280 (5)	C(5′)–O(5′)	1·432 (5)	1·425 (5)
N(7)–C(8)	1·323 (5)	1·296 (4)			
C(8)–N(9)	1·367 (5)	1·375 (4)			
C(2)–N(1)–C(6)	123·6 (3)	123·6 (3)	C(4)–N(9)–C(1′)	128·2 (3)	127·1 (3)
C(2)–N(1)–C(1)	117·8 (3)	119·7 (3)	C(8)–N(9)–C(1′)	125·8 (3)	126·7 (3)
C(6)–N(1)–C(1)	118·5 (3)	116·7 (3)	N(9)–C(1′)–C(2′)	114·5 (3)	114·3 (3)
N(1)–C(2)–N(3)	126·1 (3)	126·9 (3)	N(9)–C(1′)–O(4′)	108·0 (3)	107·6 (3)
C(2)–N(3)–C(4)	112·6 (3)	111·6 (3)	C(2′)–C(1′)–O(4′)	104·9 (3)	107·2 (3)
N(3)–C(4)–C(5)	126·8 (3)	126·3 (3)	C(1′)–C(2′)–C(3′)	102·1 (3)	101·5 (3)
N(3)–C(4)–N(9)	127·9 (3)	127·3 (3)	C(1′)–C(2′)–O(2′)	112·5 (3)	114·4 (3)
C(5)–C(4)–N(9)	105·2 (3)	106·4 (3)	C(3′)–C(2′)–O(2′)	114·7 (3)	116·6 (3)
C(4)–C(5)–C(6)	119·6 (3)	121·5 (3)	C(2′)–C(3′)–C(4′)	102·3 (3)	101·5 (3)
C(4)–C(5)–N(7)	111·5 (3)	110·6 (3)	C(2′)–C(3′)–O(3′)	110·9 (3)	107·6 (3)
C(6)–C(5)–N(7)	128·9 (3)	127·9 (3)	C(4′)–C(3′)–O(3′)	110·7 (3)	112·2 (3)
N(1)–C(6)–C(5)	111·1 (3)	110·1 (3)	C(3′)–C(4′)–C(5′)	114·3 (3)	114·9 (3)
N(1)–C(6)–N(6)	120·3 (3)	120·4 (3)	C(3′)–C(4′)–O(4′)	106·8 (3)	105·4 (3)
C(5)–C(6)–N(6)	128·6 (3)	129·5 (3)	C(5′)–C(4′)–O(4′)	109·0 (3)	109·1 (3)
C(5)–N(7)–C(8)	103·3 (3)	104·2 (3)	C(4′)–C(5′)–O(5′)	112·6 (3)	111·1 (3)
N(7)–C(8)–N(9)	113·9 (3)	113·2 (3)	C(1′)–O(4′)–C(4′)	109·4 (3)	110·1 (3)
C(4)–N(9)–C(8)	106·0 (3)	105·6 (3)			



Molecule (*a*)



Molecule (*b*)

Fig. 1. *ORTEP*II drawings (Johnson, 1976) of the independent molecules (*a*) and (*b*) with atomic numbering.

angle N(6)–C(6)–C(5) and C(2)–N(1)–C(6) are larger by *ca* 6 and 5°, respectively, whereas the angle N(1)–C(6)–C(5) is smaller by *ca* 7°. The bond distances and angles found in the sugar moiety are in good agreement with the standard values of C(2′)-*endo* puckering (Arnott & Hukins, 1972).

The molecular conformations of the two independent molecules (*a*) and (*b*) are shown in Fig. 1. Significant conformational differences were found between the two molecules. Molecule (*a*) has a *syn* conformation with a torsional angle χ_{CN} [C(4)–N(9)–C(1′)–O(4′)] of 64·1 (4)°, whereas molecule (*b*) has an *anti* conformation with χ_{CN} of −100·2 (4)°. Many crystallographic and spectroscopic studies on β-purine nucleosides have shown that both *anti* and *syn* conformers are allowed and are at equilibrium in solution (Saenger, 1984). Our results are unequivocally consistent with these data. The other examples of the coexistence of *anti* and *syn* conformations within the same crystal are found in the following crystal structures: the complex of 7-methylguanosine and its hydroiodide (Yamagata, Fukumoto, Hamada, Fujiwara & Tomita, 1983), adenosine 3′,5′-(hydrogen phosphate) (cyclic AMP) (Watenpaugh, Dow, Jensen & Furberg, 1968) and 2′-deoxy-2′-fluoroinosine (Hakoshima, Omori, Tomita, Miki & Ikehara, 1981). The conformations about the exocyclic C(4′)–C(5′) bond are also different for the two molecules. Molecule (*a*) adopts a common *gauche*-

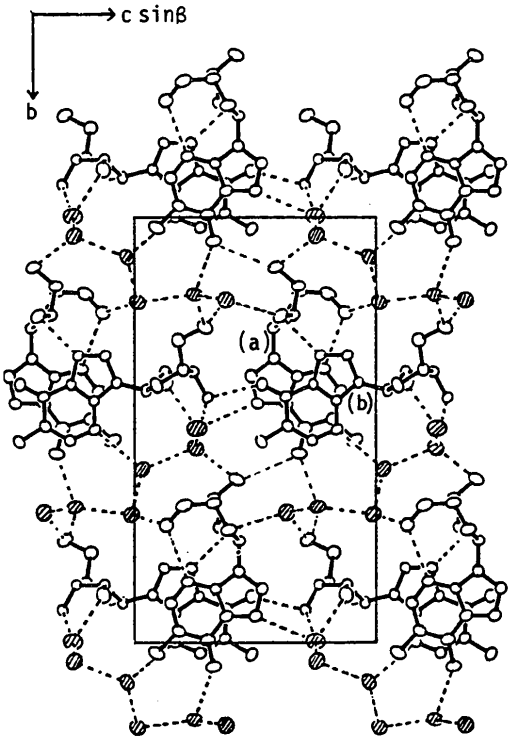


Fig. 2. Crystal structure projected down the *a* axis. Hydrogen bonds are indicated by broken lines.

gauche conformation, forming an intramolecular hydrogen bond between N(3) of the 1-methyladenine base and O(5′) of the sugar moiety with N···O 2·899 (4) Å. This type of hydrogen bond is found in many crystal structures of purine nucleosides with the *syn* conformation. On the other hand, the conformation about the C(4′)–C(5′) bond of molecule (*b*) is unusually *trans-gauche*. Both molecules have the sugar conformation as C(2′)-*endo* type. The pseudorotation parameters (Altona & Sundaralingam, 1972) are $P = 158{\cdot}4°$ (^2E), $\tau_m = 37{\cdot}4°$ for molecule (*a*) and $P = 182{\cdot}3°$ ($_3T^2$), $\tau_m = 36{\cdot}8°$ for molecule (*b*).

Fig. 2 shows the crystal structure projected down the *a* axis. The most pronounced feature of the crystal structure is the alternating parallel stacking of two independent purine bases to form columns parallel to the *a* axis. The characteristic of the stacking mode is the prominent overlapping of the pyrimidine moieties of 1-methyladenine rings. Such an extent of base overlap has not yet been observed in the unsubstituted adenine derivatives (Saenger, 1984). The base separation is about 3·4 Å and the dihedral angle 2·2 (1)°. Six crystallographically independent water molecules occupy the spaces between the columns and participate in many hydrogen bonds as shown in Fig. 2.

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Structure of Bis(thiobenzamidoethyl) Disulfide

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Abstract. $C_{18}H_{20}N_2S_4$, $M_r = 392{\cdot}61$, triclinic, $P\bar{1}$, $a = 10{\cdot}370$ (4), $b = 12{\cdot}125$ (6), $c = 8{\cdot}765$ (4) Å, $\alpha = 99{\cdot}90$ (4), $\beta = 98{\cdot}98$ (4), $\gamma = 108{\cdot}23$ (5)°, $V = 1004{\cdot}9$ (8) Å^3, $Z = 2$, $D_m = 1{\cdot}30$, $D_x = 1{\cdot}297$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 4{\cdot}56$ cm^{-1}, $F(000) = 412$, $T = 293$ K, final $R = 0{\cdot}061$ for 2291 reflections. The molecule is the dimer $PhC(S)NHCH_2CH_2S$–$SCH_2CH_2NHC(S)Ph$ bridged by an S–S bond of 2·042 Å. The torsion angle C–S–S–C is 81·5 (3)°, and the C–S–S angles are 104·3 (2) and 104·0 (2)°. The C(sp^3)–S bond lengths are 1·819 (6) and 1·813 (6) Å. The C(sp^2)–N bonds in the thioamide groups are 1·333 (6) and 1·342 (7) Å. The C=S bond lengths are 1·659 (7) and 1·681 (7) Å and related S atoms are involved in intermolecular N–H···S hydrogen bonds of 3·432 (6) and 3·531 (6) Å, respectively.

Introduction. For many years our attention has been directed towards the chemistry and biology of sulfur-containing compounds, especially amides and cor-

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