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1,9-Dimethyladeninium Chloride*

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Abstract. $C_7H_{10}N_5^+ . Cl^-$, monoclinic, $P2_1/a$, $a = 17.474$ (4), $b = 13.718$ (4), $c = 7.639$ (2) Å, $\beta = 96.03$ (2)°, $V = 1820.9$ Å³, $Z = 8$, $D_m = 1.46$ (1), $D_c = 1.46$ Mg m⁻³. Full-matrix least-squares refinement (nonhydrogen atoms anisotropic, H atoms included but not refined) based on 3439 counter-collected F_o's led to final R and weighted R values of 0.076 and 0.054 respectively. The two crystallographically independent 1,9-dimethyladeninium cations are related by a pseudo center of symmetry and form hydrogen-bonded dimers in the crystal. The exocyclic amino group of one of the cations donates a hydrogen bond to the imidazole-ring nitrogen atom N(7) of the other cation and *vice versa*. Several interactions of the type $D–H \cdots Cl^-$ are noted between the purine cations and the chloride anions.

Introduction. Chiang, Epps, Marzilli & Kistenmacher (1979) have recently determined the crystal and molecular structure of a cobalt(III) complex containing the 1,9-dimethyladeninium cation as a ligand in an

initial attempt at elucidating the metal-coordination properties of N(1)-alkylated purines. Such N(1)-alkylated purines are to be found in natural, biologically important systems, including some tRNA's (McCloskey & Nishimura, 1977; Rich, 1977). Many of these N-methylated purines are located in essentially invariant and pivotal regions of tRNA (McCloskey & Nishimura, 1977). In one important example (Rich, 1977), it was found that N(1)-methylated adenosine occurs at residue 58 in the TC loop of tRNA^{Phe} and this 1-methyladenosine residue is hydrogen bonded to a thymidine base (residue 54 in the TC loop) *via* a reversed Hoogsteen scheme (Hoogsteen, 1963). This interloop hydrogen bonding clearly plays a major role in determining the tertiary structure of tRNA^{Phe}, but it is unknown as to whether such intraloop hydrogen bonding affects the functional behavior of various tRNAs. In this paper, we present a structural analysis of the uncomplexed 1,9-dimethyladeninium cation, as the chloride salt, in the course of our studies on the effects on the molecular structural parameters in purine ligands owing to metal coordination (Kistenmacher & Marzilli, 1977) and to probe further the nature of the

intermolecular forces in crystals of nucleic acid components.

1,9-Dimethyladeninium chloride was obtained by passing 1,9-dimethyladeninium iodide (Chiang, Epps, Marzilli & Kistenmacher, 1979) through a Dowex-1-X8 ion-exchange column. Colorless crystals were obtained from a mixture of methanol and water. Preliminary oscillation and Weissenberg photographs showed the crystal system to be monoclinic with systematic absences $h0l$, $h = 2n + 1$ and $0k0$, $k = 2n + 1$ consistent with the space group $P2_1/a$. Unit-cell dimensions and their standard deviations were derived from a least-squares fit to the setting angles of 15 carefully centered reflections on a Syntex $P\bar{1}$ automated diffractometer. The crystal density, measured by neutral buoyancy methods in carbon tetrachloride and cyclohexane, indicated eight formula units per unit cell.

The 8699 reflections in the $+h$ hemisphere to $2\theta = 55°$ were surveyed on the diffractometer, with graphite-monochromatized Mo $K\alpha$ radiation. The crystal used for data collection, mounted approximately along the c axis, had the following mean separations between principal faces: (001)–(00$\bar{1}$) 0·30, (010)–(0$\bar{1}$0) 0·35, and (100)–($\bar{1}$00) 0·15 mm. Intensity data were collected in the θ–2θ scan mode with individual scan speeds (2–24° min^{-1}) determined from a rapid scan at the calculated Bragg peak. The intensities of three standards were monitored after every 100 reflections and showed no systematic variation over the course of the experiment. The measured intensities were symmetry averaged (R factor for averaging = 2·8%) and reduced to a set of 3439 independent intensities with $I \geq \sigma(I)$. Observational variances were assigned on the basis $\sigma^2(I) = S + (B_1 + B_2)(T_S/2T_B)^2 + (pI)^2$, where S, B_1, and B_2 are the scan and extrema background counts, T_S and T_B are the scan and individual background counting times ($T_B = T_S/4$ for all reflections) and p was taken to be 0·03 and represents an estimate of the error

proportional to the diffracted intensity (Busing & Levy, 1957). The intensities and their standard deviations were corrected for Lorentz and polarization effects. An absorption correction ($\mu = 0·38$ mm^{-1}) was also applied, based on the dimensions and face assignments given above, and led to maximum and minimum transmission factors of 0·95 and 0·88 respectively. An approximate absolute scale was determined by the method of Wilson (1942).

The structure was solved by direct methods employing the *MULTAN* series of programs (Germain, Main & Woolfson, 1971). All 26 nonhydrogen atoms of the two independent 1,9-dimethyladeninium chloride units were found on the E map derived from the sign set with the highest figure of merit. Several cycles of isotropic and anisotropic refinement, minimizing the quantity $\sum w(|F_o| - |F_c|)^2$ where $w = 4F_o^2/\sigma^2(F_o^2)$, led to an R value ($\sum ||F_o| - |F_c||/\sum |F_o|$) of 0·122. At this stage, a difference-Fourier map was computed and positional assignments were obtained for the 20 H atoms; the isotropic thermal parameters of the H atoms were set approximately equal to those of the atom to which they were bonded. Three further cycles of refinement, employing anisotropic thermal parameters for the non-hydrogen atoms and holding the H-atom parameters fixed, led to convergence (all shift/error values less than 0·3) and a final R value of 0·076. The final weighted R value $\{R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}\}$ and goodness of fit $\{[\sum w(|F_o| - |F_c|)^2/(NO - NV)]^{1/2}$, where $NO = 3439$ observations and $NV = 235$ variables} were 0·054 and 1·82 respectively. A final difference-Fourier map was essentially featureless.

Neutral-atom scattering-factor tables for the non-hydrogen atoms were taken from the compilation of Hanson, Herman, Lea & Skillman (1964), while those for the H atoms were from Stewart, Davidson & Simpson (1965). The scattering curves for all of the nonhydrogen atoms were corrected for anomalous-

Table 1. *Final nonhydrogen atomic coordinates*

Parameters for Cl(1) and Cl(2) are $\times 10^5$; parameters for all other atoms $\times 10^4$; estimated standard deviations in the least significant figure are enclosed in parentheses here and in succeeding tables.

	x	y	z		x	y	z
Cl(1)	24955 (4)	−15349 (5)	48892 (10)	Cl(2)	25457 (4)	8608 (5)	98913 (10)
N(1)	3742 (1)	1120 (1)	3907 (3)	N(11)	3746 (1)	3539 (1)	8931 (3)
N(3)	4606 (1)	2358 (1)	3149 (3)	N(13)	4570 (1)	4773 (1)	7977 (3)
N(6)	3922 (1)	−551 (1)	3482 (3)	N(16)	3975 (1)	1865 (1)	8689 (3)
N(7)	5427 (1)	65 (1)	2137 (3)	N(17)	5455 (1)	2483 (1)	7253 (3)
N(9)	5720 (1)	1657 (1)	1995 (3)	N(19)	5703 (1)	4069 (1)	6892 (3)
C(1)	2998 (2)	930 (2)	4608 (4)	C(11)	3014 (2)	3353 (2)	9670 (4)
C(2)	3982 (2)	2065 (2)	3749 (4)	C(12)	3951 (2)	4487 (2)	8629 (4)
C(4)	5037 (2)	1599 (2)	2664 (4)	C(14)	5019 (2)	4020 (2)	7573 (4)
C(5)	4856 (1)	631 (2)	2738 (3)	C(15)	4867 (2)	3046 (2)	7792 (4)
C(6)	4167 (1)	351 (2)	3379 (3)	C(16)	4190 (2)	2769 (2)	8469 (3)
C(8)	5938 (2)	710 (2)	1719 (4)	C(18)	5936 (2)	3126 (2)	6735 (4)
C(9)	6168 (2)	2541 (2)	1723 (4)	C(19)	6110 (2)	4955 (2)	6442 (4)

dispersion effects (Cromer & Liberman, 1970). Final atomic positional parameters for the nonhydrogen atoms are collected in Table 1.*

The structure-factor and least-squares calculations were carried out with an extensively modified version of *ORFLS* (Busing, Martin & Levy, 1962); Fourier syntheses were computed with *FORDAP* (Zalkin, 1965); the absorption correction was applied using *ORABS* (Wehe, Busing & Levy, 1962); best planes were computed with *MEAN PLANE* (Pippy & Ahmed, 1968); and illustrations were prepared with the aid of *ORTEP* (Johnson, 1965). Calculations not cited were performed with locally written programs.

Discussion. The basic molecular geometry of the 1,9-dimethyladeninium cation and the primary association between the cations and the chloride anions are illustrated in Fig. 1. A complete list of nonhydrogen-atom distances and angles as well as a comparison with those found in the complexed 1,9-dimethyladeninium ligand in [bis(acetylacetonato)(nitro)(1,9-dimethyladeninium)cobalt(III)] [bis(acetylacetonato)(dinitro)cobaltate(III)] are presented in Table 2. The molecular dimensions of the two independent 1,9-dimethyladeninium cations are in excellent internal agreement and their individual and average values are in good agreement with several N(1)-protonated adenine derivatives such as adenine hydrochloride hemihydrate (Kistenmacher & Shigematsu, 1974a), adenine dihydrochloride (Kistenmacher & Shigematsu, 1974b), and adenosine hydrochloride (Shikata, Ueki & Mitsui, 1973).

The comparison of the molecular dimensions of the two independent 1,9-dimethyladeninium cations with
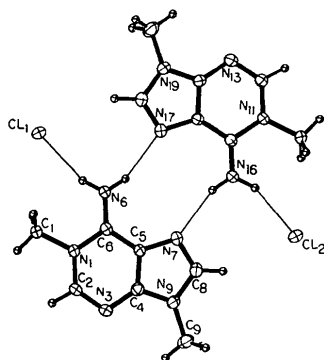
Fig. 1. Primary association of the 1,9-dimethyladeninium cations and the chloride anions in the structure of 1,9-dimethyladeninium chloride. Thin lines denote hydrogen bonds.

Table 2. *Heavy-atom interatomic distances* (Å) *and angles* (°)

	Molecule (I)	Molecule (II)	Co^III complex*
N(1)—C(2)	1·371 (4)	1·375 (3)	1·36 (1)
N(1)—C(6)	1·375 (3)	1·378 (3)	1·37 (1)
N(1)—C(1)	1·480 (4)	1·474 (4)	1·47 (1)
N(3)—C(2)	1·292 (4)	1·298 (4)	1·32 (1)
N(3)—C(4)	1·359 (4)	1·354 (4)	1·33 (1)
N(6)—C(6)	1·313 (3)	1·311 (3)	1·30 (1)
N(7)—C(5)	1·380 (3)	1·382 (4)	1·39 (1)
N(7)—C(8)	1·318 (4)	1·309 (4)	1·31 (1)
N(9)—C(4)	1·350 (4)	1·353 (4)	1·38 (1)
N(9)—C(8)	1·376 (4)	1·365 (4)	1·33 (1)
N(9)—C(9)	1·470 (4)	1·468 (4)	1·47 (1)
C(4)—C(5)	1·368 (4)	1·376 (4)	1·37 (1)
C(5)—C(6)	1·402 (4)	1·393 (4)	1·43 (1)
C(1)—N(1)—C(2)	119·1 (2)	118·6 (2)	117·8 (6)
C(1)—N(1)—C(6)	119·5 (2)	120·1 (2)	119·8 (6)
C(2)—N(1)—C(6)	121·4 (2)	121·2 (2)	122·4 (6)
C(2)—N(3)—C(4)	111·8 (2)	112·6 (2)	111·7 (6)
C(5)—N(7)—C(8)	103·5 (2)	103·5 (2)	104·4 (6)
C(4)—N(9)—C(8)	105·8 (2)	105·7 (2)	106·5 (6)
C(4)—N(9)—C(9)	127·4 (2)	126·9 (2)	124·9 (6)
C(8)—N(9)—C(9)	126·7 (2)	127·4 (2)	128·5 (6)
N(1)—C(2)—N(3)	127·1 (3)	126·3 (3)	125·8 (7)
N(3)—C(4)—N(9)	126·5 (2)	127·4 (2)	125·1 (6)
N(3)—C(4)—C(5)	126·6 (3)	126·1 (2)	129·0 (7)
N(9)—C(4)—C(5)	106·9 (2)	106·5 (2)	105·9 (6)
N(7)—C(5)—C(4)	110·8 (2)	110·4 (2)	109·3 (6)
N(7)—C(5)—C(6)	129·8 (2)	130·1 (2)	133·8 (6)
C(4)—C(5)—C(6)	119·5 (2)	119·5 (2)	116·9 (6)
N(1)—C(6)—N(6)	121·1 (2)	121·0 (2)	120·5 (6)
N(1)—C(6)—C(5)	113·7 (2)	114·1 (2)	114·0 (6)
N(6)—C(6)—C(5)	125·2 (2)	124·9 (2)	125·5 (6)
N(7)—C(8)—N(9)	113·1 (2)	113·9 (3)	113·9 (6)

* Dimensions of the 1,9-dimethyladeninium ligand in [bis-(acetylacetonato)(nitro)(1,9-dimethyladeninium)cobalt(III)] [bis-(acetylacetonato)(dinitro)cobaltate(III)] (Chiang *et al.*, 1979).
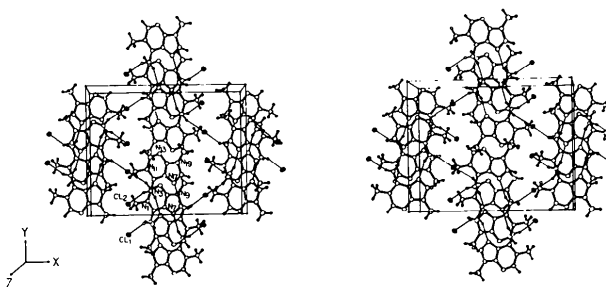

Fig. 2. A stereoview of the crystal packing in 1,9-dimethyladeninium chloride. Thin lines denote interactions of the type *D*—H···*A* (see text).

those in the N(7)-bound Co^III complex (Table 2) clearly shows the effects of the metal coordination. Upon complexation, there is a significant elongation (0·03 Å) of the N(9)—C(4), N(3)—C(2), and C(5)—C(6) bonds and a similar contraction in the N(9)—C(8) and the N(3)—C(4) bonds. Bond-angle expansions upon metal coordination at N(7) are noted in N(3)—C(4)—C(5)

Table 3. *Distances* (Å) *and angles* (°) *in the intermolecular contacts of the type* $D-H \cdots A$

D	H	A	$D-H$	$H \cdots A$	$D \cdots A$	$\angle D-H \cdots A$
N(6)	H(N6a)	Cl(1)	0·87	2·29	3·124 (2)	160
N(6)	H(N6b)	N(17^i)	0·87	2·12	2·942 (3)	158
N(16)	H(N16a)	Cl(2)	0·88	2·25	3·075 (2)	157
N(16)	H(N16b)	N(7^i)	0·86	2·12	2·939 (3)	159
C(2)	H(C2)	Cl(1^{ii})	0·96	2·54	3·464 (2)	162
C(12)	H(C12)	Cl(2^{iii})	0·96	2·58	3·506 (2)	162

Symmetry transforms

(i) $1 - x, -y, 1 - z$ (ii) $\frac{1}{2} - x, -\frac{1}{2} + y, 1 - z$ (iii) $\frac{1}{2} - x, \frac{1}{2} + y, 2 - z$

and N(7)—C(5)—C(6) while bond-angle contractions are indicated at C(4)—N(9)—C(9) and C(4)—C(5)—C(6). Similar effects owing to metal coordination at N(7) have been noted elsewhere [see Kistenmacher & Marzilli (1977), and references therein].

The nine-atom frameworks of the 1,9-dimethyl-adeninium cations are quite planar with a maximum deviation of about 0·02 Å for any one atom. A measurable fold about the C(4)—C(5) bond has been found in a variety of coordinated and uncoordinated purine fused-ring systems (Sletten & Jensen, 1969; Voet & Rich, 1970); we note, for example, that the fold about the C(4)—C(5) bond in the complexed 1,9-dimethyl-adeninium cation (Chiang *et al.*, 1979) is 2·6 (4)°. However, in the 1,9-dimethyladeninium cations described here, the folding about the C(4)—C(5) bond is minimal with dihedral angles between highly planar imidazole and pyrimidine rings of only 0·5 (2)° for the cation containing N(1) and 1·0 (2)° for the cation containing N(11). The higher degree of planarity in the uncoordinated cations as opposed to the complexed cation is surely due to the less restrictive environment about the uncoordinated purine cations and because the intermolecular interactions between the purine cations (Figs. 1 and 2) and the hydrogen-acceptor chloride anions here (Figs. 1 and 2) involve essentially coplanar cationic and anionic species.

As shown in Fig. 1, the 1,9-dimethyladeninium cations are related by a pseudo center of symmetry to form hydrogen-bond dimers in the solid. One of the protons on each of the exocyclic amino groups [N(6)H$_2$ and N(16)H$_2$] acts as a hydrogen-bond donor to the N(7) imidazole ring N atom [labeled as N(7) and N(17)] of the other cation (Table 3). The same type of hydrogen bonding has been found between residues A9 and A23 of the U12–A23 base pair in tRNA^{Phe} (Rich, 1977) and between two adenine residues in double-helical polyriboadenylic acid (Rich, Davies, Crick & Watson, 1961). The remaining proton on each of the exocyclic amino groups of the two purine cations acts as a donor in a hydrogen-bonding interaction with a chloride anion. A similar hydrogen-bonding scheme is present in the structure of adenine hydrochloride hemi-hydrate (Kistenmacher & Shigematsu, 1974a). In

addition to the hydrogen bonding involving the protons on the exocyclic amino groups, there are significant interactions of the type $C-H \cdots Cl^-$ involving the protons of the pyrimidine atoms C(2) and C(12) (Table 3 and Fig. 2), leading to interactive chains along the *a* axis. As expected for purine cations (Kistenmacher & Shigematsu, 1974a,b; Voet & Rich, 1970), there is little base-stacking overlap in the present structure (Fig. 2).

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1,3,4,5-*endo*,7,7-Hexachloro-2-azabicyclo[2.2.1]hept-2-ene: A Diels–Alder Adduct of Vinyl Chloride and 2,3,4,5,5-Pentachloro-1-azacyclopentadiene

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Abstract. $C_6H_3Cl_6N$, monoclinic, $P2_1/n$, $Z = 4$, $a = 6\cdot179$ (4), $b = 14\cdot799$ (6), $c = 11\cdot693$ (4) Å, $\beta = 92\cdot25$ (5)°, $D_c = 1\cdot876$ Mg m^{-3}. The structure was solved by direct phasing methods and refined to $R = 0\cdot051$ for 1961 independent reflections. Both bridgeheads are C–Cl. The bicyclo ring structure contains a –N=CCl moiety.

Introduction. The title compound was prepared by Gladstone, Daniels & Wong (1977) as part of a program investigating Diels–Alder reactions involving 1-azadienes.
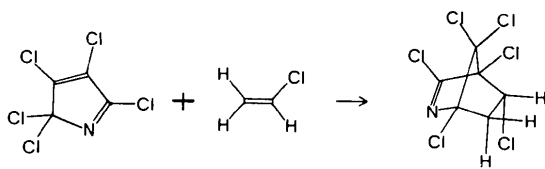
Without the heterocyclic N present, similar reactions are used to prepare the commercial insecticides aldrin and dieldrin. These reactions are both stereo- and regiospecific, yielding only the *endo*-5-substituted azanorbornenes. This work shows that in adduct (4) of the above reference (which is the title compound) the N is located at position 2, rather than the originally assigned position 1.

Translucent crystals with good face development were obtained from Gladstone, Daniels & Wong (1977). X-ray diffraction data showed the crystals to be monoclinic, with systematic absences $h0l$: $h + l = 2n$ and $0k0$: $k = 2n$, space group $P2_1/n$. The alternative space group assignment of $P2_1/c$ leads to a very large β angle.

1961 independent reflections were collected with an Enraf–Nonius CAD-4 diffractometer (Mo $K\alpha$ radiation). The crystal exhibited darkening as a result of X-ray exposure; however, three reference reflection intensities remained essentially constant through the data collection. Lorentz and polarization corrections were made in the usual manner and the data were reduced to observed structure factor amplitudes.

The phase problem was solved by symbolic addition with the program *SHELX* 76 (Sheldrick, 1976). 232 reflections with $E > 1\cdot1$ were used with a starting set of 17 reflections. An analysis for sign indeterminance in the best four solutions led to the omission of 10 reflections from the starting set. After the selection of a new starting set, two solutions were calculated, one containing a recognizable molecular fragment. A structure factor calculation on the 10 highest peaks ($R = 41\%$), followed by a difference Fourier map, was sufficient to locate the remaining three heavy atoms.

The structure was refined by the full-matrix least-squares program *ORXFLS*3 (Busing, Martin & Levy, 1971) with isotropic and then anisotropic temperature factors, until $R = 0\cdot067$. H atoms were found on a difference Fourier map and refined with isotropic temperature factors. H scattering factors were those of Stewart, Davidson & Simpson (1965). A final conventional R factor of $0\cdot051$ ($R_w = 0\cdot082$) was obtained, excluding unobserved reflections (215 reflections with $F_o < 3\sigma$). The final difference Fourier map displayed no distinguishing features.*

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34420 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.