molecules (I) and ($\bar{I} + 2c$). Therefore, BMDMTF molecules form a sheet network in the (101) plane, as seen in Fig. 2(*b*), which provides possible conductivity pathways for the electrons.

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Structure of 3-(2,6-Dichlorobenzyl)-*N*,*N*-dimethyladenine

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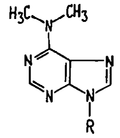
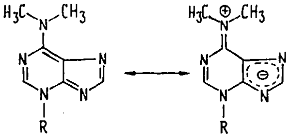
Abstract. $C_{14}H_{13}Cl_2N_5$, $M_r = 322{\cdot}20$, monoclinic, $P2_1/c$, $a = 11{\cdot}057\,(2)$, $b = 13{\cdot}802\,(2)$, $c = 9{\cdot}168\,(1)$ Å, $\beta = 91{\cdot}07\,(1)°$, $V = 1398{\cdot}9\,(3)$ Å^3, $Z = 4$, $D_x = 1{\cdot}530$ g cm^{-3}, Cu $K\alpha$ radiation (graphite monochromator), $\lambda = 1{\cdot}54051$ Å, $\mu = 4{\cdot}25$ cm^{-1}, $F(000) = 664$, room temperature, $R = 0{\cdot}039$ for 1312 observed reflections. The molecular structure has a dipolar form. The purine system is essentially planar with a short exocyclic C_{ar}—N bond of 1·331 (6) Å. The crystal packing is characterized by stacking interactions between the heterocycle and the Cl atom.

Introduction. It has been shown previously (Ramzaeva, Goldberg, Alksnis, Lídaka & Shymanska, 1989) that alkylation of *N*,*N*-dimethyladenine with benzyl halides using phase-transfer catalysis gives a mixture of *N*,*N*,9- and *N*,*N*,3-trisubstituted adenines (I) and (II).

^{1}H NMR spectra of *N*,*N*-dimethyl-3-substituted adenines (II) suggest that the CH_3 groups are nonequivalent because of their hindered rotation (Itaya, Matsumoto & Ogawa, 1980; Muravich-Alexandr, Yel'tsov & El-Sakka, 1973). This may be due to the fact that the dipolar form (III) is dominant in solution (Neiman & Bergmann, 1968). The structure of (II) was further substantiated by X-ray analysis of *N*,*N*-dimethyl-3-(2,6-dichlorobenzyl)adenine.


(I)


(II) (III)

Experimental. (II) was prepared as described by Ramzaeva, Goldberg, Alksnis, Lídaka & Shimanskaya (1989). Monocrystals were grown from absolute ethanol; crystal size 0·30 × 0·25 × 0·15 mm; lattice parameters were refined from 20 reflections with $27 \le \theta \le 32°$ on a Syntex $P2_1$ diffractometer; $\sin\theta/\lambda \le 0{\cdot}576$ Å^{-1}, $\theta/2\theta$ scan, 1904 reflections h 0–13, k 0–16, l −13–13; one standard reflection showed no significant decay, no correction for absorption.

For structure solution, initial phases of ten strong reflections were determined by the maximum-determinant method (Tsoucaris, 1980; Mishnev & Belyakov, 1988). The phase values obtained were introduced into the starting set of *MULTAN*

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters for the non-*H *atoms with e.s.d.'s in parentheses*

$$B_{\mathrm{eq}} = (B_{11}B_{22}B_{33})^{1/3}.$$

	x	y	z	B_{eq}(Å^2)
Cl(1)	0·0651 (1)	0·3613 (1)	0·2058 (1)	3·6 (1)
Cl(2)	−0·2530 (1)	0·6108 (1)	0·4480 (1)	3·6 (1)
N(1)	−0·3316 (4)	0·6750 (3)	0·0031 (4)	3·1 (2)
C(2)	−0·2425 (5)	0·6545 (3)	0·0934 (5)	2·9 (3)
N(3)	−0·1901 (3)	0·5666 (3)	0·1114 (4)	2·5 (2)
C(4)	−0·2371 (4)	0·4927 (3)	0·0270 (5)	2·3 (2)
C(5)	−0·3353 (4)	0·5097 (3)	−0·0698 (5)	2·5 (2)
C(6)	−0·3811 (4)	0·6039 (3)	−0·0837 (5)	2·8 (2)
N(7)	−0·3612 (4)	0·4229 (3)	−0·1377 (4)	3·1 (2)
C(8)	−0·2801 (5)	0·3635 (4)	−0·0771 (6)	3·6 (3)
N(9)	−0·1998 (4)	0·4010 (3)	0·0245 (4)	3·1 (2)
C(10)	−0·0822 (4)	0·5528 (4)	0·2064 (5)	2·8 (3)
C(11)	−0·1023 (4)	0·4807 (3)	0·3271 (5)	2·1 (2)
C(12)	−0·0427 (4)	0·3917 (4)	0·3357 (5)	2·7 (3)
C(13)	−0·0606 (5)	0·3243 (4)	0·4438 (5)	2·9 (3)
C(14)	−0·1410 (5)	0·3460 (4)	0·5517 (6)	3·3 (3)
C(15)	−0·2015 (5)	0·4349 (4)	0·5525 (6)	3·3 (3)
C(16)	−0·1791 (4)	0·4997 (3)	0·4404 (5)	2·6 (2)
N(17)	−0·4694 (4)	0·6327 (3)	−0·1744 (4)	3·4 (2)
C(18)	−0·5110 (6)	0·7330 (5)	−0·1799 (9)	4·7 (4)
C(19)	−0·5294 (6)	0·5665 (5)	−0·2775 (7)	4·4 (4)

Table 2. *Bond distances* (Å) *and angles* (°)

Cl(1)—C(12)	1·752 (5)	N(7)—C(8)	1·329 (7)
Cl(2)—C(16)	1·740 (5)	C(8)—N(9)	1·376 (7)
N(1)—C(2)	1·306 (6)	C(10)—C(11)	1·507 (7)
N(1)—C(6)	1·372 (6)	C(11)—C(12)	1·395 (6)
C(2)—N(3)	1·354 (6)	C(11)—C(16)	1·379 (6)
N(3)—C(4)	1·376 (6)	C(12)—C(13)	1·376 (7)
N(3)—C(10)	1·476 (6)	C(13)—C(14)	1·376 (7)
C(4)—C(5)	1·409 (6)	C(14)—C(15)	1·398 (7)
C(4)—N(9)	1·332 (6)	C(15)—C(16)	1·388 (7)
C(5)—C(6)	1·399 (7)	N(17)—C(18)	1·459 (8)
C(5)—N(7)	1·378 (6)	N(17)—C(19)	1·465 (8)
C(6)—N(17)	1·331 (6)		

C(2)—N(1)—C(6)	120·1 (4)	N(3)—C(10)—C(11)	113·0 (4)
N(1)—C(2)—N(3)	126·0 (4)	C(10)—C(11)—C(12)	123·2 (4)
C(2)—N(3)—C(4)	116·1 (4)	C(10)—C(11)—C(16)	122·0 (4)
C(2)—N(3)—C(10)	121·8 (4)	C(12)—C(11)—C(16)	114·9 (4)
C(4)—N(3)—C(10)	122·0 (4)	Cl(1)—C(12)—C(11)	119·4 (4)
N(3)—C(4)—C(5)	120·6 (4)	Cl(1)—C(12)—C(13)	115·9 (4)
N(3)—C(4)—N(9)	126·9 (4)	C(11)—C(12)—C(13)	124·2 (4)
C(5)—C(4)—N(9)	112·5 (4)	C(12)—C(13)—C(14)	118·4 (4)
C(4)—C(5)—C(6)	119·0 (4)	C(13)—C(14)—C(15)	120·7 (5)
C(4)—C(5)—N(7)	106·9 (4)	C(14)—C(15)—C(16)	118·0 (5)
C(6)—C(5)—N(7)	134·0 (4)	Cl(2)—C(16)—C(11)	119·6 (4)
N(1)—C(6)—C(5)	118·2 (4)	Cl(2)—C(16)—C(15)	116·6 (4)
N(1)—C(6)—N(17)	115·4 (4)	C(11)—C(16)—C(15)	123·8 (4)
C(5)—C(6)—N(17)	126·5 (4)	C(6)—N(17)—C(18)	122·2 (5)
C(5)—N(7)—C(8)	102·4 (4)	C(6)—N(17)—C(19)	122·3 (4)
N(7)—C(8)—N(9)	118·3 (5)	C(18)—N(17)—C(19)	115·5 (5)
C(4)—N(9)—C(8)	99·9 (4)		

(Germain, Main & Woolfson, 1971). Two variants were calculated, one of them yielding the model. For structure refinement, 1312 independent reflections with $I > 2\sigma(I)$ were used, Fourier synthesis, full-matrix least squares (F^2), $w^{-1} = \sigma^2(F)$, anisotropic thermal parameters for C, Cl and N, isotropic for H, located from difference synthesis, $R = 0{\cdot}039$, $wR = 0{\cdot}039$ [$I > 2\sigma(I)$]; for all data $R = 0{\cdot}049$, $wR = 0{\cdot}044$; max. $\Delta/\sigma = 0{\cdot}65$; max. deviations in final ΔF synthesis within $\pm 0{\cdot}2$ e Å^{-3}. Calculations carried out with the Syntex (1976) *XTL/XTLE* system. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Discussion. Table 1 lists the atomic coordinates of the molecule and their equivalent isotropic thermal parameters.* Fig. 1 shows a perspective view of the molecular structure. Table 2 gives the values of bond lengths and valence angles. Features of the crystal packing in the unit cell are shown in Fig. 2.

Generally, the purine system in nucleosides does not assume a planar conformation (Mishnev & Bleidelis, 1984). In the title molecule the dihedral angle between the pyrimidine and imidazole planes is 1·2° and the purine system is essentially planar, as observed in the related *N*,*N*-dimethyladenine (Dahl, 1987) and *N*-methyl-9-(2-fluorophenyl)adenine (Lisgarten & Palmer, 1988). The equation of the

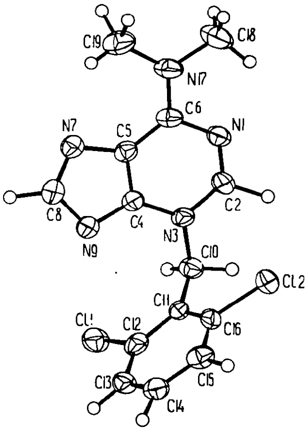

Fig. 1. Perspective view of the molecule.

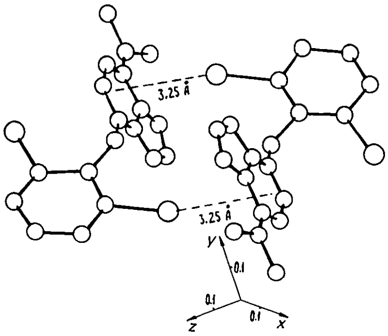

Fig. 2. Intermolecular contacts between Cl and the purine system in the crystal structure.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53126 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

least-squares plane defined by the atoms N(1), C(2), N(3), C(4), C(5), C(6), N(7), C(8) and N(9) is $0·661x + 0·214y - 0·719z + 0·458 = 0$, where x, y, z are Cartesian coordinates in Å. Atoms C(5) and C(6) have the maximum deviations from the plane of −0·018 (4) and +0·018 (5) Å, respectively, and the displacements of C(10) and N(17) are 0·104 (5) and 0·066 (4) Å, respectively.

The phenyl ring is planar. Its normal equation is $-0·737x - 0·409y - 0·539z + 3·467 = 0$. The dihedral angle between this plane and the purine system is 100·8°. The torsion angles C(4)—N(3)—C(10)—C(11) and N(3)—C(10)—C(11)—C(12) are −64·0 (5) and 113·6 (5)°, respectively.

The C(6)—N(17) bond length is short at 1·331 (6) Å. By comparison, the C_{ar}—NH_2 bond in aniline is 1·402 Å (Lister, Tyler, Hog & Larsen, 1974), whilst its length in *N*,*N*-dimethyladenine is 1·355 Å (Dahl, 1987). Consequently, form (III) describes the observed structure most accurately and confirms the correct interpretation of the NMR spectral data. The geometry of the five-membered ring suggests delocalization of the negative charge over all five atoms. However, the length of the C(5)—C(4), C(5)—C(6) and C(5)—N(7) bonds [1·409 (6), 1·399 (7) and 1·378 (6) Å] may be indicative of higher electron density at C(5).

Stacking interactions in the crystal structure are observed between the Cl atom and the purine system (Fig. 2). The distance from the Cl atom to the heterocyclic plane is 3·25 Å. All other intermolecularic contacts correspond to sums of van der Waals radii (Zefirov & Zorkii, 1976).

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Triphenylphosphine: a Redetermination

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Abstract. $C_{18}H_{15}P$, $M_r = 262·3$, monoclinic, $P2_1/a$, $a = 11·329$ (3), $b = 14·915$ (4), $c = 8·440$ (1) Å, $\beta = 92·12$ (2)°, $V = 1\,425$ (1) $Å^3$, $Z = 4$, $D_x = 1·22$ g cm^{-3}, Mo $K\alpha$, $\bar{\lambda} = 0·71069$ Å, $\mu = 1·70$ cm^{-1}, $T = 200$ K, $F(000) = 552$, $R = 0·038$ for 2135 unique observed reflections. The crystal structure of the title compound has been redetermined, to improved precision, using diffractometer data at reduced temperature. The average P—C distance is 1·831 Å, and C—P—C angle is 102·8° (e.s.d.'s on individual parameters being 0·002 Å and 0·1°, respectively). The improved precision allows identification of several distortions, such as C—C bond-length variations, in the phenyl rings.

Introduction. Triphenylphosphine, PPh_3, and its derivatives have found widespread use as ligands in transition-metal chemistry, notably in homogeneous catalysis. The single-crystal X-ray structure of triphenylphosphine was reported in 1964 (Daly, 1964). As part of a general study on the geometric deformations in tertiary phosphine complexes (Orpen & Connelly, 1990; Dunne & Orpen 1990) precise values for the mean P—C bond length and C—P—C bond angle in the uncomplexed ligand were required. The structure determined by Daly using photographic techniques, with the visual estimation of reflection intensities, was refined to a crystallographic R factor of 0·101. Thus, there existed a need for a (low-

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