atom attached, while the other does not (Craven, McMullan, Bell & Freeman, 1977). In order to compare the effects of substituents on the bond distances in 2-amino-4,5-dicyanoimidazole it is necessary to average the non-equivalent bonds in the low-temperature imidazole structure. Also comparison is best made with the bond distances uncorrected for thermal motion since no such corrections have been made in the present work. There are no significant differences in bond angles between the two structures. The average N(3)—C(4) bond distance of 1·384 (2) Å is significantly longer than the value of 1·365 Å reported for imidazole, and similarly the C(4)—C(4') distance of 1·372 (4) Å is significantly longer than the imidazole value of 1·359 Å. This may be due to the electron-withdrawing tendency of the cyano groups at this end of the molecule.

The high decomposition temperature of this compound may be due to the six hydrogen bonds formed by each molecule with its neighbors as illustrated in Fig. 2. There are strong hydrogen bonds of length 2·756 (2) Å between N(3) atoms in different molecules. Each molecule forms two weaker hydrogen bonds from amino N—H to cyano groups in neighboring molecules, of length 3·195 Å, and two additional non-equivalent hydrogen bonds from the cyano groups to the N—H groups in neighboring molecules, of length 3·127 Å. The extension of this pattern forms sheets of molecules in the (202) crystal planes. This result is in agreement with the observed

infrared spectrum of the solid (Rasmussen, 1984) which shows several peaks in the N—H region. In order to accommodate this pattern of hydrogen bonding, both molecules A and B are bent significantly out of plane with C(4) 0·05 and 0·04 Å respectively, from the least-squares plane. In comparison, imidazole forms only two N—H···N hydrogen bonds per molecule (Craven et al., 1977).

The crystal packing is shown in Fig. 3.

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Structure of the 2:1 Complex of 2-[4,5-Bis(methylthio)-1,3-dithiol-2-ylidene]-1,3-dithiolo[4,5-d]dithiole (BMDMTF) and 7,7',8,8'-Tetracyano-p-quinodimethane (TCNQ)*

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Abstract. BMDMTFNTCNQ (2/1), $2C_9H_8S_8.C_{12}H_4N_4$, $M_r = 949\cdot46$, triclinic, $P\bar{1}$, $a = 8\cdot570$ (3), $b = 10\cdot098$ (4), $c = 12\cdot217$ (4) Å, $\alpha = 98\cdot97$ (3), $\beta = 77\cdot67$ (3), $\gamma = 107\cdot14$ (3)°, $V = 982\cdot09$ Å^3, $D_x = 1\cdot603$ g cm^{-3}, $Z = 1$, $\lambda(Cu\,K\alpha) = 1\cdot5418$ Å, $\mu = 80\cdot84$ cm^{-1}, $F(000) = 484$, $T = 295$ K, $R = 5\cdot5\%$ ($wR = 4\cdot3\%$) for 2373 observed reflections. The generally planar BMDMTF (D) and TCNQ (A) molecules follow each other in ADDADD order in the [$\bar{1}$20] direction with their molecular planes mutually arranged in a common plane parallel to (211); the

* TCNQ is 2,2'-(2,5-cyclohexadiene-1,4-diylidene)bispropanedinitrile.

molecular planes of neighboring rows are in the same common plane, and these flat layers build the structure. There are S⋯S contacts between BMDMTF molecules in and between the layers forming infinite corrugated chains and a sheet network parallel to the (101) plane.

Introduction. The title compound belongs to a group of novel organic conductors based on the unsymmetrical π donor bis(methylthio)(methylenedithio)-tetrathiafulvalene (Fig. 1a), which is derived from the tetrathiafulvalene (TTF) backbone.

Since the discovery of superconductivity and antiferromagnetism in the organic Bechgaard salts (Thorup, Rindorf, Soling & Bechgaard, 1981), intensive research has been devoted to the synthesis of new π-donor molecules. The salts obtained generally form structures characterized by columns of these molecules which allow for quasi one-dimensional conductivity pathways via S⋯S contacts. In order to introduce coupling between these conducting pathways, several unsymmetrical derivatives of TTF were synthesized (Coulon, Amiel, Chasseau, Manhal & Fabre, 1986; Granier, Gallois & Fabre, 1989; Mentzafos, Psycharis & Terzis, 1989; Saito, Ishikawa, Kikuchi, Ikemoto & Kobayashi, 1989). The BMDMTF–TCNQ complex has recently been synthesized. The salt, however, is a semiconductor. The purpose of this paper is to establish the crystallographic structure of the title compound and to compare the packing with related complexes.

Experimental. A mixture of BMDMTF and TCNQ was dissolved in methyl cyanide and heated. After slow cooling of the solution, crystals of the title complex were obtained. A black plate-shaped crystal, size $0.05 \times 0.2 \times 0.6$ mm, was mounted on a DEC Micro PDP computer-controlled Stoe four-circle diffractometer. Lattice constants from least-squares refinement of 38 reflections ($36 < 2\theta < 50°$). The conventional cell is $a = 8.570$ (3), $b = 12.217$ (4), $c = 10.098$ (4) Å, $\alpha = 77.67$ (3), $\beta = 72.86$ (3), $\gamma = 81.03$ (3)°, which is transformed by matrix (100, 00−1, 010). Intensity data in a hemisphere h 0 to 14, k −17 to 17, l −20 to 20 in the range $5 < 2\theta < 120°$ were measured in the ω–2θ scan mode (Ni-filtered Cu $K\alpha$ radiation). A variable scan range, $\Delta\theta = (1.1 + 0.26\tan\theta)°$, was used with minimum and maximum scan speeds of 0.01 and 0.04° s⁻¹. Three standard reflections measured every 15 reflections showed no significant variation. 2917 unique reflections, 544 unobserved reflections [$I < 2\sigma(I)$, $R_{int} = 0.032$]. Data corrected for Lorentz–polarization effects. A numerical correction for absorption was applied; T_{max}/T_{min}: 0.447/0.030.

The structure was solved by direct methods using SHELXS86 (Sheldrick, 1985). Structure completed

Table 1. *Final positional and thermal parameters* ($\text{Å}^2 \times 10^2$) *with e.s.d.'s in parentheses*

	x	y	z	U_{eq}/U
(a) BMDMTF				
S(1)	0.0870 (3)	0.1287 (2)	0.9042 (1)	8.16 (8)
S(2)	−0.0278 (2)	0.3664 (2)	0.8702 (1)	7.87 (7)
S(3)	0.3288 (2)	0.1611 (2)	0.6779 (1)	5.20 (6)
S(4)	0.2160 (2)	0.4012 (1)	0.6449 (1)	4.62 (5)
S(5)	0.3764 (2)	0.3710 (1)	0.3812 (1)	4.61 (5)
S(6)	0.4914 (2)	0.1344 (1)	0.4142 (1)	4.69 (5)
S(7)	0.4845 (3)	0.3354 (2)	0.1290 (1)	10.8 (1)
S(8)	0.6072 (2)	0.0709 (2)	0.1665 (1)	5.57 (6)
C(1)	0.006 (1)	0.273 (1)	0.9730 (6)	8.4 (4)
C(2)	0.1804 (7)	0.2136 (6)	0.7837 (4)	4.6 (2)
C(3)	0.1283 (7)	0.3197 (6)	0.7674 (4)	4.5 (2)
C(4)	0.3166 (6)	0.2711 (5)	0.5846 (4)	3.9 (2)
C(5)	0.3862 (6)	0.2596 (5)	0.4742 (4)	3.6 (2)
C(6)	0.4640 (7)	0.2798 (6)	0.2614 (4)	4.8 (2)
C(7)	0.5124 (7)	0.1724 (6)	0.2750 (4)	4.4 (2)
C(8)	0.396 (1)	0.4762 (8)	0.1566 (6)	10.0 (4)
C(9)	0.4305 (9)	−0.0756 (6)	0.1430 (5)	7.3 (3)
H(1A)	−0.095 (8)	0.234 (6)	1.020 (6)	10 (3)
H(1B)	0.091 (7)	0.333 (6)	0.995 (5)	6 (3)
H(8A)	0.267 (1)	0.4437 (8)	0.1952 (6)	33 (7)
H(8B)	0.462 (1)	0.5586 (8)	0.2110 (6)	24 (6)
H(8C)	0.408 (1)	0.5106 (8)	0.0749 (6)	20 (4)
H(9A)	0.3934 (9)	−0.1462 (6)	0.2176 (5)	15 (3)
H(9B)	0.3332 (9)	−0.0463 (6)	0.1438 (5)	7 (2)
H(9C)	0.4666 (9)	−0.1285 (6)	0.0618 (5)	16 (3)
(b) TCNQ				
N(1)	0.0382 (8)	0.6982 (6)	0.7446 (5)	8.7 (3)
N(2)	0.2279 (7)	0.6277 (6)	0.3871 (5)	8.9 (3)
C(21)	−0.0416 (7)	0.9652 (6)	0.6107 (5)	4.1 (2)
C(22)	0.0803 (7)	0.9246 (6)	0.4116 (5)	4.2 (2)
C(23)	0.0410 (7)	0.8820 (5)	0.5243 (4)	4.0 (2)
C(24)	0.0804 (7)	0.7692 (5)	0.5466 (5)	4.9 (2)
C(25)	0.0528 (8)	0.7293 (6)	0.6586 (6)	6.0 (2)
C(26)	0.1630 (8)	0.6886 (6)	0.4588 (5)	5.7 (2)
H(21)	−0.072 (6)	0.930 (5)	0.674 (4)	4 (2)
H(22)	0.144 (6)	0.869 (5)	0.362 (4)	5 (2)

by the Fourier difference method and refined subsequently with XTAL (Hall & Stewart, 1987). All non-H atoms refined with anisotropic displacement parameters. H atoms of TCNQ and methylene in BMDMTF were located from a difference synthesis and refined with isotropic displacement parameters. Methyl groups were treated as rigid groups with normal sp^3 geometry (C—H = 1.08 Å). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 149). After convergence, $R = 5.5\%$ and $wR = 4.3\%$ for 2373 observed reflections, $w = 1/\sigma^2(F)$, $S = 2.7$, total number of refined parameters 248. $(\Delta/\sigma)_{max} = 0.0005$, $(\Delta/\sigma)_{av} = 0.0001$, largest peak in final difference map $= 0.71$, largest hole $= 0.64$ e Å⁻³, near S(7).*

**Discussion.** Fractional coordinates of the title compound are listed in Table 1. The atomic numbering

scheme used for BMDMTF is given in Fig. 1(a) and for TCNQ in Fig. 1(b). Bond lengths and angles are listed in Tables 2 and 3.

Complex conformations. In the BMDMTF donor molecule, all bond lengths and angles of the TTF backbone are similar to those found in other TTF derivatives (Galigné, Liautard, Peytavin, Brun, Maurin, Fabre, Torreilles & Giral, 1979a,b; Galigné, Peytavin, Liautard & Brun, 1980).

The geometry of the BMDMTF molecule can be described by three major planes. Plane I consists of the atoms S(1), S(2), C(2), C(3), S(3) and S(4) with an average out-of-plane distance from the least-squares plane of 0·02 Å. C(1) is 0·52 (1) Å away from plane I. Plane II is defined by part of the fulvalene moiety [atoms S(3), S(4), S(5), S(6), C(4) and C(5)], showing an average out-of-plane distance from the least-squares plane of 0·03 Å. Plane III is made up by the five-membered ring C(5), S(5), C(6), C(7), S(6) plus S(7), C(8) and S(8). The average distance of these atoms from the least-squares plane is 0·01 Å. The terminal C(8) methyl group lies in plane III in a *cis* position with respect to S(5); the torsion angle is − 1·9 (4)° for S(5)—C(6)—S(7)—C(8). The second terminal methyl group C(9) points away from plane III [distance of C(9) from plane III 1·778 (7) Å]; the torsion angle with respect to S(6) is 86·3 (4)°.
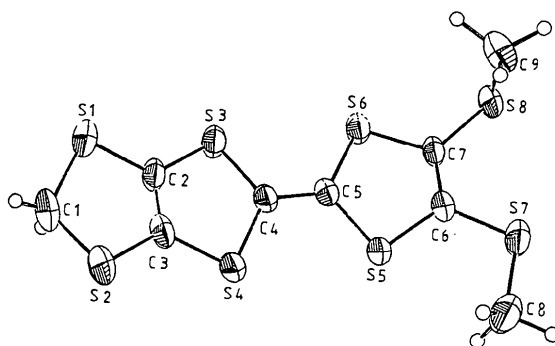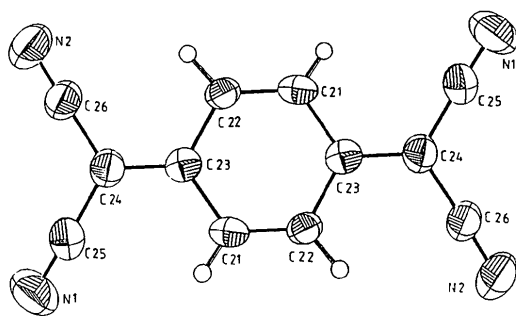
Table 2. *Bond lengths* (Å) *with e.s.d.'s in parentheses*

(a) BMDMTF

S(1)—C(1)	1·81 (1)	S(1)—C(2)	1·740 (5)
S(2)—C(1)	1·80 (1)	S(2)—C(3)	1·749 (6)
S(3)—C(2)	1·746 (5)	S(3)—C(4)	1·749 (6)
S(4)—C(3)	1·738 (5)	S(4)—C(4)	1·763 (6)
S(5)—C(5)	1·749 (6)	S(5)—C(6)	1·753 (5)
S(6)—C(5)	1·747 (6)	S(6)—C(7)	1·761 (5)
S(7)—C(6)	1·751 (6)	S(7)—C(8)	1·76 (1)
S(8)—C(7)	1·754 (5)	S(8)—C(9)	1·816 (6)
C(2)—C(3)	1·332 (9)	C(4)—C(5)	1·354 (6)
C(6)—C(7)	1·315 (9)		

(b) TCNQ

N(1)—C(25)	1·114 (9)	N(2)—C(26)	1·132 (8)
C(21)—C(23)	1·441 (8)	C(21)—C(22)	1·33 (1)
C(22)—C(23)	1·447 (8)	C(23)—C(24)	1·360 (9)
C(24)—C(25)	1·438 (9)	C(24)—C(26)	1·435 (8)

Table 3. *Valence angles* (°) *with e.s.d.'s in parentheses*

(a) BMDMTF

C(1)—S(1)—C(2)	92·4 (3)	S(4)—C(3)—C(2)	118·4 (4)
C(1)—S(2)—C(3)	92·6 (4)	S(3)—C(4)—S(4)	115·6 (3)
C(2)—S(3)—C(4)	93·2 (3)	S(3)—C(4)—C(5)	121·7 (4)
C(3)—S(4)—C(4)	92·8 (3)	S(4)—C(4)—C(5)	122·6 (5)
C(5)—S(5)—C(6)	94·3 (3)	S(5)—C(5)—S(6)	115·1 (3)
C(5)—S(6)—C(7)	94·5 (3)	S(5)—C(5)—C(4)	121·8 (4)
C(6)—S(7)—C(8)	104·6 (3)	S(6)—C(5)—C(4)	123·1 (5)
C(7)—S(8)—C(9)	100·7 (3)	S(5)—C(6)—S(7)	119·0 (4)
S(1)—C(1)—S(2)	110·4 (4)	S(5)—C(6)—C(7)	118·3 (4)
S(1)—C(2)—S(3)	124·0 (4)	S(7)—C(6)—C(7)	122·7 (4)
S(1)—C(2)—C(3)	118·1 (4)	S(6)—C(7)—S(8)	117·6 (4)
S(3)—C(2)—C(3)	117·8 (4)	S(6)—C(7)—C(6)	117·1 (4)
S(2)—C(3)—S(4)	124·0 (4)	S(8)—C(7)—C(6)	125·2 (4)
S(2)—C(3)—C(2)	117·6 (4)		

(b) TCNQ

C(23)—C(21)—C(22)	121·9 (5)	C(23)—C(24)—C(25)	122·8 (5)
C(23)—C(22)—C(21)	121·5 (5)	C(23)—C(24)—C(26)	120·9 (5)
C(21)—C(23)—C(22)	116·5 (6)	C(25)—C(24)—C(26)	116·3 (6)
C(21)—C(23)—C(24)	122·1 (5)	N(1)—C(25)—C(24)	177·0 (8)
C(22)—C(23)—C(24)	121·3 (5)	N(2)—C(26)—C(24)	177.7 (8)

The three planes form dihedral angles of 16·91 (4) and 6·64 (4)° for planes I and II and planes II and III, respectively. This gives the whole molecule a slight U shape by which it grasps the central part of the TCNQ molecule in the neighboring layer. Another BMDMTF molecule, located in the third layer, does the same from the opposite side.

The TCNQ acceptor molecule is planar with an inversion-center symmetry. The maximum out-of-plane distance is 0·06 Å [for N(1)]; the average distance of the atoms from the plane is 0·03 Å.

Complex packing. The stoichiometry of the title complex is 2:1 for BMDMTF (D) and TCNQ (A). Plane III of BMDMTF and the TCNQ plane form a dihedral angle of 4·1 (7)° and are arranged in a common layer parallel to the crystallographic (211) plane. The in-plane packing is *ADDADD* along the [120] direction (see Fig. 2a).

Fig. 2(a) is a stereoview of the crystal packing in two directions looking at the layers from the side. Two situations are observed between adjacent layers (see Fig. 3). One is a crosswise *AD* overlap with an

(a)

(b)

Fig. 1. An *ORTEP* (Johnson, 1971) representation of the complex showing the atomic numbering scheme for (a) BMDMTF and (b) TCNQ.

interlayer distance of 3·349 (6) Å between TCNQ and the BMDMTF plane III. Another overlap is an approximate head-to-tail back-to-back *DD* overlap with an interlayer distance of 3·155 (5) Å between planes III of the two molecules.

There are short intermolecular S···S distances (shown in Table 4) not only within the layers through the S atoms of molecules (I) and (Ī + *a* + *c*),

but also between the layers through the S atoms of molecules (I) and (Ī + *a* + *b* + *c*), forming a donor BMDMTF infinite corrugated chain along the [010] direction. As the S atoms S(1) and S(2) of the terminal methylenedithio groups are not in plane III of BMDMTF, the intermolecular S···S contacts between BMDMTF chains are *via* the S(1) atoms of

Table 4. *Selected intermolecular contact distances for BMDMTF (Å)*

S3···S8ⁱ	3·463 (3)
S3···S6ⁱ	3·670 (3)
S3···C9(CH3)ⁱ	3·68 (1)
S6···S6ⁱ	3·732 (2)
C8(CH3)···C8(CH3)ⁱ	3·871 (9)
S5···S4ⁱⁱ	3·552 (2)
S1···S1ⁱⁱⁱ	3·548 (3)

The symmetry operations of the second atom in each pair are the following: (i) 1−*x*, −*y*, 1−*z*; (ii) 1−*x*, 1−*y*, 1−*z*; (iii) −*x*, −*y*, 2−*z*.
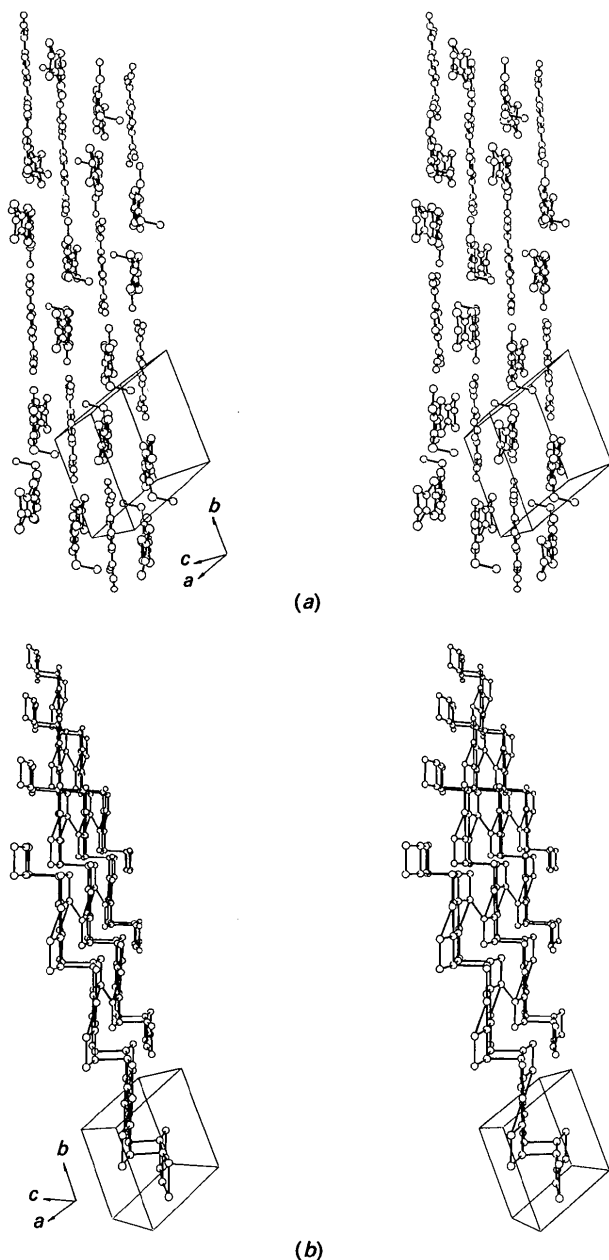

Fig. 2. (*a*) Stereoview of the title complex projected along the central double bond C(4)—C(5). (*b*) Stereoview (*SCHAKAL86*; Keller, 1986) of the BMDMTF donors. For clarity only the S atoms are shown and bond sticks represent S···S contacts.
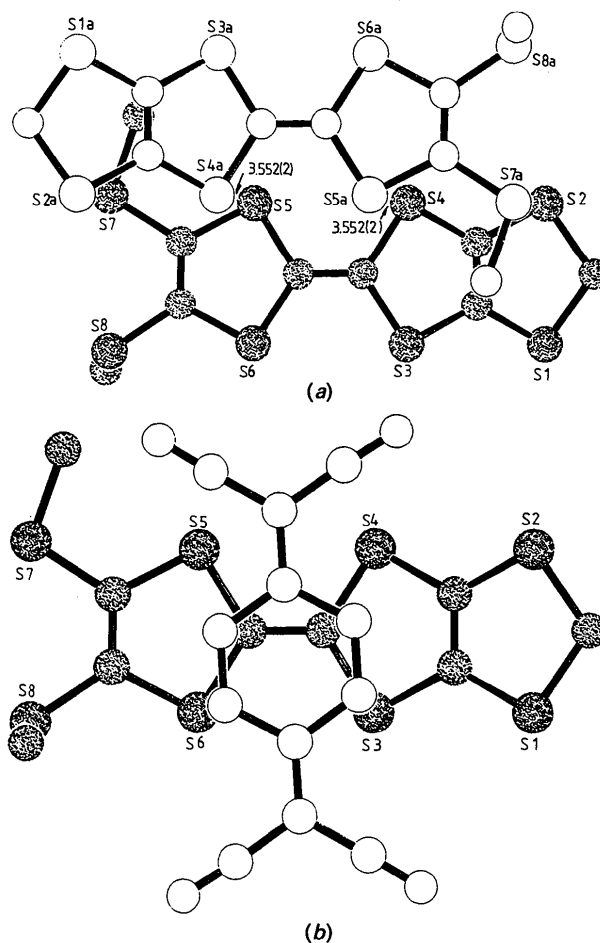

Fig. 3. Illustration of complex overlap in neighboring layers perpendicular to a layer: (*a*) BMDMTF and BMDMTF; (*b*) TCNQ and BMDMTF. Distances are in Å.

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. $C_{14}H_{13}Cl_2N_5$, $M_r = 322.20$, monoclinic, $P2_1/c$, $a = 11.057$ (2), $b = 13.802$ (2), $c = 9.168$ (1) Å, $\beta = 91.07$ (1)°, $V = 1398.9$ (3) Å^3, $Z = 4$, $D_x = 1.530$ g cm^{-3}, Cu $K\alpha$ radiation (graphite monochromator), $\lambda = 1.54051$ Å, $\mu = 4.25$ cm^{-1}, $F(000) = 664$, room temperature, $R = 0.039$ 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)
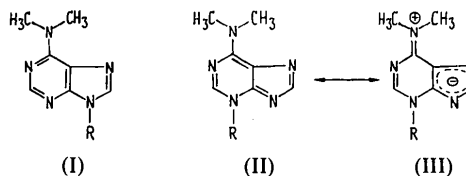
Experimental. (II) was prepared as described by Ramzaeva, Goldberg, Alksnis, Lídaka & Shimanskaya (1989). Monocrystals were grown from absolute ethanol; crystal size 0.30 × 0.25 × 0.15 mm; lattice parameters were refined from 20 reflections with $27 \le \theta \le 32°$ on a Syntex $P2_1$ diffractometer; $\sin\theta/\lambda \le 0.576$ 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*