Table 3. *Intermolecular hydrogen bonds and other significant short contacts* (Å)

Hydrogen bonds

D	A	Symmetry	$D \cdots A$	$H \cdots A$	$\angle D—H \cdots A$ (°)
(at x,y,z)					
C(2)	O(4)N*	x, y, z	3·161 (5)	2·29 (4)	147 (3)
O(5γ)	N(1')	−1 + x, ½ − y, ½ + z	2·966 (4)	2·16 (5)	178 (5)
N(4'α)	N(3')	−x, −y, 1 − z	3·011 (4)	2·13 (4)	173 (4)
N(4'α)	O(4)N*	−1 + x, y, z	3·033 (4)	2·23 (5)	168 (5)

Short contacts (*A* at x,y,z)

A	B	Symmetry	
S(1)	O(5γ)	−x, −y, 2 − z	3·246 (3)
S(1)	O(3)N	x, y, z	2·967 (4)
C(2)	O(2)N	−1 + x, y, z	3·276 (6)
C(2)	C(4')	x, y, z	3·382 (5)
C(2)	O(3)N	x, y, z	2·978 (5)
C(4)	O(4)N	−1 + x, y, z	3·363 (5)
C(4)	O(2)N	1 − x, ½ + y, ½ − z	3·302 (5)
C(4α)	O(3)N	−x, ½ + y, ½ − z	3·291 (5)
C(4α)	O(2)N	1 − x, ½ + y, ½ − z	3·235 (6)
C(5α)	C(2')	x, ½ − y, ½ + z	3·473 (5)
C(5α)	N(3')	x, ½ − y, ½ + z	3·475 (5)
C(35')	O(2)N	1 − x, ½ + y, ½ − z	3·165 (6)
C(4')	N(3)	x, y, z	3·296 (4)
N(4'α)	O(2)N	−1 + x, y, z	3·056 (5)
C(5')	C(2)	x, y, z	2·811 (5)
C(6')	O(4)N	x, y, z	3·333 (5)
C(6')	O(3)N	1 − x, ½ + y, ½ − z	3·300 (5)

* The suffix *N* indicates the nitrate ion.

to consider it to be a hydrogen bond, and a corresponding hydrogen bond has been observed in many other thiamine structures (Richardson, Franklin & Thompson, 1975). Furthermore, the nitrate ions participate in the relatively strong short contacts (less than

3·1 Å) of $O(3) \cdots S(1)$, $O(3) \cdots C(2)$ and $O(2) \cdots N(4'\alpha)$ atomic pairs. These interactions, in addition to an $N(4'\alpha) \cdots N(3')$ hydrogen bond, contribute to the stabilization of the molecular packing in the *a* and *b* directions.

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Structure of 9-Ethyl-7-methylguaninium Iodide Dimethyl Sulfoxide Solvate, C₈H₁₂N₅O⁺.I⁻.C₂H₆OS

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Abstract. $M_r = 399·25$, monoclinic, $P2_1/c$, $a = 10·266$ (2), $b = 6·976$ (1), $c = 22·059$ (4) Å, $\beta = 97·66$ (1)°, $V = 1565·8$ (4) Å³, $Z = 4$, $D_m = 1·734$ (5), $D_x = 1·693$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1·5418$ Å, $\mu(\text{Cu } K\alpha) = 17·65$ mm⁻¹, $F(000) = 796$, $T = 293$ K. Final $R = 0·085$ for 2659 independent reflections. The molecular dimensions, especially in the imidazole moiety, are significantly different from those of neutral guanine. The crystal structure consists of extensive overlapping of the centrosymmetrically related guaninium rings.

Introduction. 7-Methylguaninium is a biologically important nucleic acid base forming the 'cap' structure of most eukaryotic mRNA's (Shatkin, 1976). The presence of this base, in the form of m⁷GpppN(m), has been shown to be necessary for the efficient binding of mRNA to ribosome and the translation of the mRNA to proteins (Tamura, Imae & Strominger, 1976; Spratt & Strominger, 1976; Weber, Hickey, Nuss & Baglioni, 1977). On the other hand, the main site of DNA attacked by the mutagenic alkylating agents is the N(7) atom of guanine base (Brookes & Lawley, 1964). Elucidation of the structural properties of N(7)-alkylated guanine and comparison with the neutral molecule may be useful in gaining a more complete understanding of the above-mentioned biological phenomena. We have therefore determined the crystal structure of 9-ethyl-7-methylguaninium (MEG) iodide.

Experimental. Synthesis by the reaction of 9-methyl-guanine with methyl iodide, following the published procedure (Jones & Robins, 1963); crystallization from dimethyl sulfoxide (0·3 M) as platelets; crystal 0·2 × 0·2 × 0·1 mm sealed in a glass capillary tube ($\varnothing$ = 0·5 mm) in the presence of some mother liquid; Rigaku automated four-circle diffractometer, graphite-monochromated Cu $K\alpha$ radiation; cell parameters determined by least-squares fit to 2θ and -2θ values of 24 reflections ($45° \leq 2\theta \leq 55°$); D_m measured by flotation in CCl_4/C_6H_6 mixture; intensity data within $2\theta = 130°$ collected by ω–2θ scan mode with scan speed $4°(2\theta)\text{min}^{-1}$, scan width (2θ) $(1·2 + 0·15\tan\theta)°$, background 5 s (both sides of each peak) at 40kV and 150mA; h 0–12, k 0–8, l −26–26; 2659 [2464 with $I > \sigma(I)$] independent reflections measured; three reflections monitored at 100-reflection intervals showed no intensity deterioration; Lorentz and polarization corrections applied, experimental absorption correction ignored; structure solved by heavy-atom method, refined by block-diagonal least squares with anisotropic thermal parameters; although most H atoms could be located from a difference Fourier map, all except the H atoms of the C(11) methyl group were included in their calculated idealized positions as a fixed contribution; final $R = 0·085$ and $R_w = 0·118$, respectively; $\sum w(|F_o|-|F_c|)^2$ minimized, $w = 0·08573$ for $F_o = 0·0$, $w = 1·0/[\sigma(F_o)^2 -0·09762\ |F_o| +0·00916\ |F_o|^2]$ for $F_o > 0·0$; a final difference map showed residual fluctuations of $-1·25$ to $0·85$ e Å^{-3} (especially close to the iodide ion); all Δ/σ for non-H atoms <1 in final cycle; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); all numerical calculations made on an ACOS-900 computer at the Computation Center of Osaka University using *The Universal Crystallographic Computing System* (1979).*

Discussion. Final atomic parameters of non-H atoms are listed in Table 1, and interatomic distances and angles with e.s.d.'s are in Table 2. An *ORTEP* (Johnson, 1965) drawing of the molecule, with atom-numbering scheme, is presented in Fig. 1. Although the e.s.d.'s for the bonding parameters are relatively large because of the effect of the iodide ion, they clearly show the structural features of an N(7)-protonated guanine (Taylor & Kennard, 1982): enlargements of C(5)–N(7)–C(8) and C(8)–N(9)–C(4) bond angles, reductions of N(7)–C(8)–N(9) and C(4)–C(5)–N(7) bond angles and shortening of C(8)–N(9) bond length compared with the standard values for neutral guanine

Table 1. *Atomic coordinates for non-H atoms with their e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{4}{3}(B_{11}a^2+B_{22}b^2+B_{33}c^2+acB_{13}\cos\beta).$$

	x	y	z	$B_{eq}(\text{Å}^2)$
I	0·68886 (5)	0·1830 (1)	0·64537 (2)	4·49
N(1)	0·9422 (6)	0·275 (1)	1·0447 (3)	3·08
C(2)	0·8625 (7)	0·238 (1)	0·9899 (3)	2·66
N(2)	0·7328 (6)	0·235 (1)	0·9924 (3)	4·08
N(3)	0·9089 (6)	0·2053 (9)	0·9380 (3)	3·22
C(4)	1·0386 (7)	0·215 (1)	0·9437 (4)	3·16
C(5)	1·1249 (7)	0·251 (1)	0·9969 (4)	3·05
C(6)	1·0767 (8)	0·282 (1)	1·0525 (4)	3·47
O(6)	1·1390 (6)	0·3113 (9)	1·1034 (3)	4·53
N(7)	1·2503 (6)	0·242 (1)	0·9800 (3)	3·55
C(7)	1·3734 (9)	0·269 (2)	1·0221 (5)	5·67
C(8)	1·2424 (8)	0·200 (1)	0·9216 (5)	4·46
N(9)	1·1164 (7)	0·189 (1)	0·8990 (3)	4·26
C(10)	1·063 (1)	0·131 (2)	0·8351 (5)	5·61
C(11)	1·038 (5)	0·282 (5)	0·7956 (9)	33·86
S(1)	0·5416 (2)	0·1826 (3)	0·8282 (1)	3·87
O(1)S	0·5317 (6)	0·182 (1)	0·8961 (3)	4·81
C(2)S	0·651 (1)	−0·002 (2)	0·8156 (5)	5·19
C(3)S	0·646 (1)	0·384 (2)	0·8163 (5)	5·41

Table 2. *Bond lengths (Å) and angles (°) between non-H atoms*

|			
N(1)–C(2)	1·39 (1)	N(1)–C(6)	1·37 (1)
C(2)–N(2)	1·34 (1)	C(2)–N(3)	1·32 (1)
N(3)–C(4)	1·32 (1)	C(4)–C(5)	1·40 (1)
C(4)–N(9)	1·36 (1)	C(5)–C(6)	1·40 (1)
C(5)–N(7)	1·39 (1)	C(6)–O(6)	1·23 (1)
N(7)–C(7)	1·48 (2)	N(7)–C(8)	1·31 (1)
C(8)–N(9)	1·33 (1)	N(9)–C(10)	1·50 (2)
C(10)–C(11)	1·37 (5)	S(1)–O(1)S	1·516 (7)
S(1)–C(2)S	1·76 (1)	S(1)–C(3)S	1·81 (1)
C(2)–N(1)–C(6)	125·7 (7)	N(1)–C(2)–N(2)	116·1 (7)
N(1)–C(2)–N(3)	123·2 (7)	N(2)–C(2)–N(3)	120·7 (7)
C(2)–N(3)–C(4)	112·6 (7)	N(3)–C(4)–C(5)	127·5 (8)
N(3)–C(4)–N(9)	127·2 (8)	C(5)–C(4)–N(9)	105·4 (7)
C(4)–C(5)–C(6)	120·4 (7)	C(4)–C(5)–N(7)	106·0 (7)
C(6)–C(5)–N(7)	133·6 (8)	N(1)–C(6)–C(5)	110·6 (7)
N(1)–C(6)–O(6)	120·9 (8)	C(5)–C(6)–O(6)	128·5 (8)
C(5)–N(7)–C(7)	124·9 (8)	C(5)–N(7)–C(8)	109·5 (8)
C(7)–N(7)–C(8)	125·5 (9)	N(7)–C(8)–N(9)	108·3 (9)
C(4)–N(9)–C(8)	110·8 (8)	C(4)–N(9)–C(10)	122·8 (8)
C(8)–N(9)–C(10)	126·0 (9)	N(9)–C(10)–C(11)	114 (2)
O(1)S–S(1)–C(2)S	106·3 (5)	O(1)S–S(1)–C(3)S	105·4 (5)
C(2)S–S(1)–C(3)S	98·2 (5)		
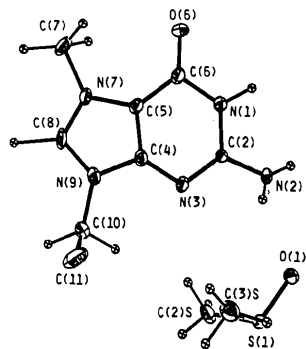
Fig. 1. *ORTEP* drawing of the MEG cation and dimethyl sulfoxide, along with the atomic numbering. The thermal ellipsoids correspond to 50% probability [20% for C(11)].
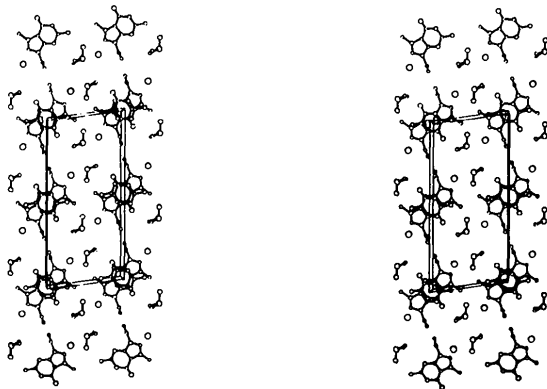
Fig. 2. A stereoscopic view of the crystal structure, viewed along **b**.

Fig. 3. Extensive overlapping mode of the neighbouring MEG molecules, viewed (a) perpendicular and (b) parallel to the central guaninium ring.

[104·2 (3), 106·0 (2), 113·5 (4) and 110·8 (2)°, and 1·374 (4) Å, respectively].

The displacements of the ring and substituted atoms from the best-fit plane of the guaninium ring show no significant difference from those of the neutral base, atomic fluctuations ranging from −0·10 (2) [C(10)] to 0·013 (9) Å [C(4)] and the dihedral angle between the pyrimidine and imidazole ring moieties being 1·0 (5)°. C(11) has a significantly high thermal parameter ($B_{iso} = 33·86$ Å^2), probably due to the relatively loose crystal packing in the vicinity of this atom, and lies approximately perpendicular to the ring [torsion angles C(4)—N(9)—C(10)—C(11) and C(8)—N(9)—C(10)—C(11) 95 (3) and −93 (3)°, respectively].

The crystal packing, as viewed down **b**, is shown in Fig. 2. A feature of the packing is the layer formation consisting of the prominent base stacking. It is not clear at present whether or not this extensive overlapping results from the N(7) methylation. The stacking mode is shown in Fig. 3. The interplanar spacing between the centrosymmetrically related (and therefore parallel-arranged) rings is 3·43 Å for the upper pair and 3·39 Å for the lower, which is very close to the normal van der
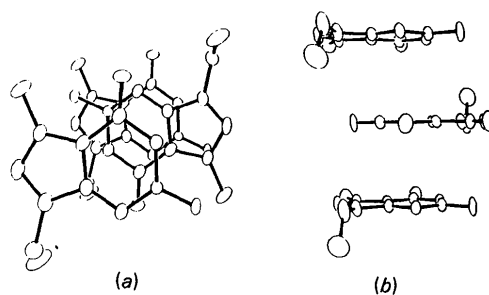
Waals separation distance (3·4 Å). In addition to three possible hydrogen bonds [N(2)$(x,y,z)\cdots$O(1)$S(x,y,z)$ = 2·78 (1) Å, N(2)$(x,y,z)\cdots$I$(x, 0·5−y, 0·5+z)$ = 3·511 (8) Å and C(8)$(x,y,z)\cdots$O(1)$S(1+x,y,z)$ = 3·10 (1) Å], these stacking layers are stabilized by Coulombic [I$(x,y,z)\cdots$C(7)$(−1+x, 0·5−y, −0·5+z)$ = 3·96 (1) Å, $\cdots$N(1)$(x, 0·5−y, −0·5+z)$ = 3·650 (7) Å and $\cdots$C(8)$(2−x, −0·5+y, 1·5−z)$ = 3·78 (1) Å] and van der Waals contact forces.

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Bis(hexahydro-1-pyridazinyl)thiophosphorsäure-O-phenylester, C14H23N4OPS

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Abstract. $M_r = 326·403$. Crystallizes from *n*-hexane in two modifications. (*A*): orthorhombic, $P2_12_12_1$, $a = 15·877$ (2), $b = 11·843$ (2), $c = 8·923$ (1) Å, $V = 1677·8$ Å^3, $Z = 4$, $D_x = 1·292$ g cm^{-3}, $F(000) = 696$, $\mu = 25·89$ cm^{-1}, $R = 0·033$ for 1595 unique contribut-

ing reflections. (*B*): triclinic, $P\bar{1}$, $a = 15·551$ (2), $b = 8·768$ (1), $c = 6·446$ (1) Å, $\alpha = 93·63$ (2), $\beta = 104·40$ (2), $\gamma = 95·52$ (1)°, $V = 843·9$ Å^3, $Z = 2$, $D_x = 1·284$ g cm^{-3}, $F(000) = 348$, $\mu = 25·73$ cm^{-1}, $R = 0·034$ for 2713 unique contributing reflections. For