C(3)–N(4)–C(5)–C(10) is 46·0 (4)°. The corresponding torsion angle in 5-(1,2-diphenylguanidino)-3H-1,2,4-dithiazole-3-thione (Butler, Glidewell & Liles, 1978) is 46·7°, but those in diphenylguanidine (Zakharov, Andrianov & Struchkov, 1980) vary from 22 to 179°. The ester group |C(20), C(21), O(22), O(23) and C(24)| is planar within 0·07 Å, and the carbamoyl group |C(24), C(25), O(26), N(27), C(28) and C(29)| is also planar within 0·005 Å. The torsion angles about the covalent bonds linking these planar groups are $-73\cdot4$ (4)° for C(16)–C(17)–C(20)–C(21), $-64\cdot5$ (3)° for C(17)–C(20)–C(21)–O(22), 85·3 (3)° for C(21)–O(23)–C(24)–C(25) and 166·4 (4)° for O(23)–C(24)–C(25)–N(27).

The intermolecular interactions around the guanidyl group are illustrated in Fig. 2. The guanidyl group forms five intermolecular hydrogen bonds; the N···O distances are 2·900 (4) Å for N(1)···O(33) (x, $\frac{3}{2}-y$, $-\frac{1}{2}+z$), 3·807(3)Å for N(1)···O(34), 2·802(2)Å for N(2)···O(26) $1+x$, y, z), 3·182(4)Å for N(2)···O(32) (x, $\frac{3}{2}-y$, $-\frac{1}{2}+z$), and 2·801 (3) Å for N(4)···O(32). The average H···O distance and N–H···O angle are 2·18 (4) Å and 163 (2)°, respectively. The topological pattern of these hydrogen bonds is the same as those found in guanidyl carboxylate and guanidyl phosphate derivatives (Salunke & Vijayan, 1981). The hydrogen-bond system is stabilized by coupling of the planar guanidyl group with the two methanesulfonate anions.

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Structure of a Cyclopropyldiaminosulfonium Salt

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Abstract. |N-(p-Chlorophenyl)-N-methyl|(morpholino)|2-(p-nitrophenyl)cyclopropylamino|oxosulfonium tetrafluoroborate, |C$_{20}$H$_{23}$ClN$_3$O$_4$S||BF$_4$|, M_r = 523·5, orthorhombic, $P2_12_12_1$, $a = 17\cdot693$ (3), $b = 9\cdot141$ (1), $c = 14\cdot768$ (3) Å, $V = 2388$ (1) Å^3, $Z = 4$, $D_x = 1\cdot456$, D_m(flotation in aqueous AgNO$_3$) = 1·446 (3) Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ = 0·303 mm^{-1}, $F(000) = 1080$, $T = 296$ K, $R = 0\cdot061$, $wR = 0\cdot072$, 2393 observations. This compound is a racemate in solution but the single crystal is optically active.

Introduction. It is well known that oxosulfonium ylides react with carbonyl compounds to give corresponding epoxides (Corey & Chaykovsky, 1965; Trost & Melvin, 1975). However, the reactivity of heteroatom-substituted oxosulfonium ylides is quite different from those of normal oxosulfonium ylides (Chalkley, Snodin, Stevens & Whiting, 1978; Shimagaki, Tsuchiya, Ban & Oishi, 1978). In previous papers, we reported that aminoaryloxosulfonium ylides react with carbonyl compounds to give β-aryloxyalkylsulfonamides and vinylsulfonamides *via* four-membered

Table 1. *Final atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\text{Å}^2 \times 10^2$), *with e.s.d.'s in parentheses*

$$B_{eq} = \tfrac{1}{3}\sum_i\sum_j B_{ij}a_i^* a_j^* \mathbf{a}_i\cdot\mathbf{a}_j.$$

	x	y	z	B_{eq}	Occupancy
S	-3190 (1)	-1762 (1)	-6808 (1)	354 (5)	1·0
Cl	344 (1)	1117 (3)	-6865 (2)	921 (13)	1·0
N1	-2406 (2)	-2550 (5)	-7056 (3)	409 (21)	1·0
N2	-3691 (2)	-3085 (5)	-6442 (3)	401 (20)	1·0
N3	-419 (3)	4456 (6)	-4719 (4)	510 (25)	1·0
O1	-3584 (2)	-1039 (4)	-7516 (3)	454 (18)	1·0
O2	-4604 (3)	-5258 (5)	-5701 (4)	685 (25)	1·0
O3	-280 (2)	4333 (6)	-3916 (4)	625 (23)	1·0
O4	-91 (3)	5329 (6)	-5208 (3)	701 (27)	1·0
C1	-2931 (3)	-561 (6)	-5945 (4)	363 (23)	1·0
C2	-3480 (3)	639 (7)	-5719 (5)	512 (30)	1·0
C3	-2781 (3)	1009 (5)	-6243 (4)	356 (23)	1·0
C4	-2137 (3)	1842 (6)	-5834 (4)	359 (22)	1·0
C5	-1802 (4)	2931 (6)	-6352 (4)	446 (26)	1·0
C6	-1233 (3)	3804 (7)	-5985 (4)	444 (27)	1·0
C7	-999 (3)	3528 (6)	-5117 (4)	381 (24)	1·0
C8	-1288 (3)	2413 (7)	-4600 (4)	429 (26)	1·0
C9	-1867 (3)	1560 (6)	-4967 (4)	425 (25)	1·0
C10	-1724 (3)	-1646 (6)	-7031 (4)	381 (23)	1·0
C11	-1239 (3)	-1835 (7)	-6308 (4)	427 (26)	1·0
C12	-593 (3)	-967 (7)	-6264 (4)	501 (30)	1·0
C13	-444 (3)	-7 (7)	-6933 (5)	532 (31)	1·0
C14	-930 (4)	152 (7)	-7667 (4)	523 (31)	1·0
C15	-1571 (3)	-658 (7)	-7709 (4)	454 (28)	1·0
C16	-2427 (4)	-3728 (7)	-7747 (4)	525 (30)	1·0
C17	-4525 (3)	-2999 (7)	-6536 (5)	553 (32)	1·0
C18	-4827 (4)	-4535 (8)	-6515 (5)	668 (39)	1·0
C19	-3805 (4)	-5371 (8)	-5664 (6)	656 (38)	1·0
C20	-3438 (3)	-3909 (7)	-5647 (5)	529 (31)	1·0
B1	-1993 (3)	-2792 (9)	-3793 (5)	600 (42)	1·0
F1A	-1823 (4)	-3039 (8)	-2958 (4)	565 (34)	0·5
F2A	-1873 (5)	-3754 (11)	-4410 (5)	889 (51)	0·5
F3A	-2033 (9)	-1546 (11)	-4079 (8)	1365 (83)	0·5
F4A	-2799 (6)	-3265 (15)	-3563 (6)	1072 (66)	0·5
F1B	-2536 (6)	-1834 (13)	-3871 (8)	1108 (67)	0·5
F2B	-1362 (7)	-2057 (22)	-4204 (8)	1588 (103)	0·5
F3B	-1560 (5)	-2999 (11)	-3021 (6)	815 (49)	0·5
F4B	-2094 (9)	-4000 (12)	-4307 (10)	1431 (87)	0·5

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

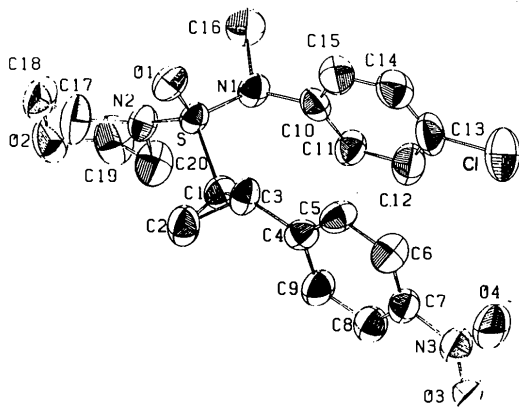|			
S-N1	1·605 (4)	C1-C2	1·503 (8)
S-N2	1·594 (4)	C1-C3	1·525 (7)
S-O1	1·421 (4)	C2-C3	1·497 (8)
S-C1	1·744 (5)	C3-C4	1·498 (7)
Cl-C13	1·736 (7)	C4-C5	1·388 (8)
N1-C10	1·463 (7)	C4-C9	1·391 (7)
N1-C16	1·484 (7)	C5-C6	1·395 (8)
N2-C17	1·484 (8)	C6-C7	1·371 (8)
N2-C20	1·465 (8)	C7-C8	1·372 (8)
N3-O3	1·216 (7)	C8-C9	1·397 (8)
N3-O4	1·223 (7)	C10-C11	1·381 (8)
N3-C7	1·456 (7)	C10-C15	1·375 (8)
O2-C18	1·427 (9)	C11-C12	1·393 (9)
O2-C19	1·419 (9)	C12-C13	1·348 (9)
		C13-C14	1·391 (9)
		C14-C15	1·355 (8)
		C17-C18	1·502 (10)
		C19-C20	1·486 (10)
N1-S-N2	102·6 (2)	C3-C4-C5	117·8 (5)
N1-S-O1	117·8 (2)	C3-C4-C9	122·6 (4)
N1-S-Cl	102·9 (2)	C5-C4-C9	119·6 (5)
N2-S-O1	109·2 (2)	C4-C5-C6	120·3 (5)
N2-S-Cl	112·1 (2)	C5-C6-C7	118·4 (5)
O1-S-Cl	112·0 (2)	N3-C7-C6	118·9 (5)
S-N1-C10	117·0 (3)	N3-C7-C8	118·1 (5)
S-N1-C16	117·4 (3)	C6-C7-C8	123·0 (5)
C10-N1-C16	116·6 (4)	C7-C8-C9	118·2 (5)
S-N2-C17	118·8 (4)	C4-C9-C8	120·4 (5)
S-N2-C20	119·4 (3)	N1-C10-C11	117·5 (4)
C17-N2-C20	114·0 (4)	N1-C10-C15	121·0 (4)
O3-N3-O4	122·8 (5)	C11-C10-C15	121·5 (5)
O3-N3-C7	118·8 (5)	C10-C11-C12	118·4 (5)
O4-N3-C7	118·4 (5)	C11-C12-C13	119·8 (6)
C18-O2-C19	110·0 (5)	Cl-C13-C12	120·1 (5)
S-C1-C2	116·9 (4)	Cl-C13-C14	118·7 (5)
S-C1-C3	115·3 (3)	C12-C13-C14	121·2 (6)
C2-C1-C3	59·2 (3)	C13-C14-C15	119·8 (6)
C1-C2-C3	61·1 (4)	C10-C15-C14	119·3 (5)
C1-C3-C2	59·7 (3)	N2-C17-C18	107·6 (5)
C1-C3-C4	119·6 (4)	O2-C18-C17	110·6 (6)
C2-C3-C4	122·4 (5)	O2-C19-C20	111·7 (6)
		N2-C20-C19	108·4 (5)

Fig. 1. View of the title compound. Thermal ellipsoids are at 50% probability (*ORTEP*II, Johnson, 1976).

cyclic intermediates (Okuma, Nakanishi & Ohta, 1984; Yokomori, Sekido, Hirokawa, Okuma & Ohta, 1985). The authors have recently found that the reaction of diaminooxosulfonium ylides with aromatic aldehydes did not give normal epoxides (Okuma, Nakanishi, Honda, Ohta, Yokomori & Sekido, 1985). The structure of the products might be cyclopropyloxosulfonium salts, but this could not be shown by spectroscopic methods. To confirm the structure and the reaction mechanism, the authors have undertaken to determine the structure of this compound by X-ray crystallography.

Experimental. Colorless cubic crystals by slow evaporation of an aqueous methanol solution at room temperature. Crystal $0.3 \times 0.3 \times 0.3$ mm, Rigaku–Denki AFC-III automated diffractometer. Systematic absences $h00$ for h odd, $0k0$ for k odd, $00l$ for l odd. Cell constants by least squares using 20 reflections with $20° < 2\theta(\text{Mo}) < 40°$ measured on the diffractometer. Intensity data collected in ω–2θ scan mode. 2395 independent reflections, $2\theta < 50.0°$, $0 \leq h \leq 20$, $0 \leq k \leq 10$, $0 \leq l \leq 17$. No systematic fluctuations in three standard reflections (542, 533, 124) monitored at the beginning and every 50 reflections. Structure determined using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Block-diagonal least-squares refinement (*HBLS*VI: Ashida, 1981), $\sum w(|F_o| - k|F_c|)^2$ minimized. Weighting scheme: $w = [\sigma^2(F_o) + 0.0714|F_o| + 0.0006|F_o|^2]^{-1}$. Subsequent difference Fourier maps revealed positions for all 23 H atoms; however, all H atoms were fixed at calculated positions (with C–H $= 1.08$ Å) with fixed isotropic temperature factors ($B = 5.0$ Å^3). Final cycle

of block-diagonal least squares gave $R = 0.061$, $wR = 0.072$, $S = 1.18$, for 2393 reflections with $|F_o| > 2\sigma(F_o)$ and 344 variables. The tetrafluoroborate anion is disordered, two possible orientations for the anion being found on a difference Fourier map. No evidence for extinction. $(\Delta/\sigma)_{max} = 0.001$. Final difference map contained no peak higher than $0.5\,e\,\text{Å}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. The final atomic coordinates with their estimated standard deviations are given in Table 1.* The numbering scheme is presented in Fig. 1. The bond distances and angles are shown in Table 2.

This compound is a racemate in solution (from circular dichroism) but the single crystal is polar.

The length of the $S^1-N(sp^2)$ bond is 1.589 (6), 1.597 (6) Å, which is in good agreement with that of the S—N bond from other experimental data (Yokomori *et al.*, 1985; Eliopoulos, Sheldrick & Hamodrakas, 1983; Cameron, Hair & Morris, 1973, 1974; Kálmán, 1967; Kálmán, Duffin & Kucsman, 1971; Jordan, Smith, Lohr & Lipscomb, 1963). The $S^1-C(sp^3)$ bond length is also in good agreement with those of other compounds (Yokomori *et al.*, 1985; Wheatley, 1954; Bullough & Wheatley, 1957; Truter, 1962).

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Study of Short Hydrogen Bonds. III.* Structures of Pyrrolidinium Hydrogen Bis(p-methylbenzoate) and Hexamethyleneiminium† Hydrogen Bis(p-methylbenzoate)

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Abstract. Pyrrolidinium hydrogen bis(p-methylbenzoate) (1), $C_4H_{10}N^+.C_{16}H_{15}O_4$, $M_r = 343.42$, orthorhombic, space group $Pcan$, $a = 9.835$ (1), $b = 25.143$ (2), $c = 7.6448$ (4) Å, $V = 1890.4$ (3) Å^3, $Z = 4$, $D_x = 1.207$ Mg m^{-3}, $\mu = 0.64$ mm^{-1}, $F(000) = 736$, final $R = 0.089$ for 1515 unique reflections. Hexamethyleneiminium hydrogen bis(p-methylbenzoate) (2), $C_6H_{14}N^+.C_{16}H_{15}O_4$, $M_r = 371.48$,

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