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3-Methyl-4,5-diphenyl-1,2,3-thiadiazolium Fluorosulphate

BY PETER G. JONES AND OLGA KENNARD*

University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW, England

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Abstract. $C_{15}H_{13}N_2S^+.FO_3S^-$, $M_r = 352{\cdot}41$, monoclinic, $C2/c$, $a = 32{\cdot}233$ (8), $b = 15{\cdot}058$ (8), $c = 14{\cdot}546$ (4) Å, $\beta = 110{\cdot}61$ (2)°, $U = 6567$ Å^3, $Z = 16$, $D_x = 1{\cdot}425$ g cm^{-3}, μ(Mo $K\alpha$) = 3·0 cm^{-1}. There are two molecules in the asymmetric unit; the heterocyclic rings show no significant differences. The anions exhibit different types of disorder. The structure was refined to $R = 0{\cdot}083$ for 5022 unique reflexions.

Table 1. *Atom coordinates* (×10^4)

The overall isotropic temperature factor for H atoms is 0·108 (6) Å^2.

	x	*y*	*z*
S(2)	3208 (1)	2527 (1)	2703 (1)
O(1)	3171 (2)	2318 (3)	3616 (3)
O(2)	3609 (1)	2264 (4)	2609 (3)
O(3)	2843 (1)	2119 (4)	1917 (4)
O(4)	3139 (2)	3474 (3)	2530 (3)
S(3)	0	950 (1)	2500
O(5)	−440 (3)	1139 (9)	2416 (9)
O(6)	222 (5)	1691 (9)	2365 (10)
F(7)	201 (3)	740 (9)	3629 (7)
O(8)	45 (7)	179 (8)	2086 (11)
S(4)	0	4637 (1)	2500
O(9)	203 (4)	3940 (8)	2186 (9)
O(10)	265 (5)	5413 (9)	2561 (11)
F(11)	96 (4)	4488 (10)	3596 (7)
O(12)	−427 (3)	4766 (11)	2072 (11)
S(1*A*)	4511 (1)	3500 (1)	3201 (1)
N(2*A*)	4232 (1)	4211 (4)	3555 (3)
N(3*A*)	3934 (1)	4594 (3)	2786 (2)
C(4*A*)	3922 (1)	4300 (3)	1888 (3)
C(5*A*)	4239 (1)	3666 (3)	1985 (3)
C(6*A*)	3638 (2)	5263 (4)	2966 (4)
C(11*A*)	3584 (1)	4621 (3)	981 (3)
C(12*A*)	3556 (2)	5492 (3)	710 (3)
C(13*A*)	3228 (2)	5775 (4)	−120 (4)
C(14*A*)	2930 (2)	5184 (5)	−692 (4)
C(15*A*)	2955 (2)	4287 (4)	−451 (3)
C(16*A*)	3287 (1)	4003 (3)	396 (3)
C(21*A*)	4359 (1)	3206 (3)	1228 (3)
C(22*A*)	4461 (2)	2312 (4)	1319 (4)
C(23*A*)	4589 (2)	1916 (5)	617 (5)
C(24*A*)	4624 (2)	2370 (5)	−163 (5)
C(25*A*)	4529 (1)	3257 (5)	−248 (3)
C(26*A*)	4394 (1)	3690 (4)	452 (3)
S(1*B*)	4260 (1)	2300 (1)	5568 (1)
N(2*B*)	4357 (1)	1706 (3)	4749 (3)
N(3*B*)	4089 (1)	1028 (3)	4533 (3)
C(4*B*)	3797 (1)	953 (3)	5010 (3)
C(5*B*)	3848 (1)	1653 (3)	5649 (3)
C(6*B*)	4132 (2)	389 (5)	3798 (5)
C(11*B*)	3491 (1)	196 (3)	4842 (3)
C(12*B*)	3116 (2)	137 (3)	4009 (4)
C(13*B*)	2839 (2)	−595 (4)	3901 (5)
C(14*B*)	2928 (2)	−1229 (4)	4593 (5)
C(15*B*)	3297 (3)	−1183 (4)	5400 (5)
C(16*B*)	3580 (2)	−454 (3)	5545 (4)
C(21*B*)	3594 (1)	1858 (3)	6280 (3)
C(22*B*)	3804 (2)	2050 (4)	7266 (3)
C(23*B*)	3556 (2)	2264 (5)	7836 (4)
C(24*B*)	3104 (2)	2310 (4)	7425 (4)
C(25*B*)	2894 (2)	2133 (4)	6450 (4)
C(26*B*)	3134 (1)	1895 (3)	5875 (3)
H(12*A*)	3796	5961	1159
H(13*A*)	3205	6468	−325
H(14*A*)	2669	5414	−1347
H(15*A*)	2718	3820	−918
H(16*A*)	3316	3310	601
H(22*A*)	4439	1935	1937
H(23*A*)	4664	1215	682
H(24*A*)	4727	2035	−707
H(25*A*)	4559	3627	−862
H(26*A*)	4317	4391	385
H(12*B*)	3039	648	3450
H(13*B*)	2545	−651	3247
H(14*B*)	2703	−1780	4502
H(15*B*)	3375	−1712	5938
H(16*B*)	3869	−404	6211
H(22*B*)	4161	2033	7589
H(23*B*)	3719	2398	8615
H(24*B*)	2913	2487	7878
H(25*B*)	2538	2181	6128
H(26*B*)	2966	1736	5105

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* External Staff, Medical Research Council.

Table 2. *Bond lengths* (Å)

N(2*A*)–S(1*A*)	1·595 (6)	N(2*B*)–S(1*B*)	1·600 (6)
C(5*A*)–S(1*A*)	1·683 (5)	C(5*B*)–S(1*B*)	1·684 (5)
N(3*A*)–N(2*A*)	1·318 (6)	N(3*B*)–N(2*B*)	1·302 (7)
C(4*A*)–N(3*A*)	1·360 (6)	C(4*B*)–N(3*B*)	1·353 (7)
C(6*A*)–N(3*A*)	1·473 (9)	C(6*B*)–N(3*B*)	1·475 (9)
C(5*A*)–C(4*A*)	1·367 (7)	C(5*B*)–C(4*B*)	1·373 (7)
C(11*A*)–C(4*A*)	1·462 (5)	C(11*B*)–C(4*B*)	1·470 (7)
C(21*A*)–C(5*A*)	1·458 (7)	C(21*B*)–C(5*B*)	1·458 (8)
C(12*A*)–C(11*A*)	1·362 (7)	C(12*B*)–C(11*B*)	1·376 (7)
C(16*A*)–C(11*A*)	1·387 (6)	C(16*B*)–C(11*B*)	1·366 (7)
C(13*A*)–C(12*A*)	1·359 (8)	C(13*B*)–C(12*B*)	1·393 (9)
C(14*A*)–C(13*A*)	1·355 (9)	C(14*B*)–C(13*B*)	1·339 (10)
C(15*A*)–C(14*A*)	1·390 (10)	C(15*B*)–C(14*B*)	1·343 (9)
C(16*A*)–C(15*A*)	1·381 (6)	C(16*B*)–C(15*B*)	1·395 (10)
C(22*A*)–C(21*A*)	1·381 (8)	C(22*B*)–C(21*B*)	1·376 (7)
C(26*A*)–C(21*A*)	1·375 (8)	C(26*B*)–C(21*B*)	1·389 (6)
C(23*A*)–C(22*A*)	1·362 (11)	C(23*B*)–C(22*B*)	1·372 (10)
C(24*A*)–C(23*A*)	1·357 (12)	C(24*B*)–C(23*B*)	1·368 (9)
C(25*A*)–C(24*A*)	1·367 (12)	C(25*B*)–C(24*B*)	1·358 (8)
C(26*A*)–C(25*A*)	1·397 (9)	C(26*B*)–C(25*B*)	1·366 (9)
O(1)–S(2)	1·403	O(2)–S(2)	1·401
O(3)–S(2)	1·453	O(4)–S(2)	1·452
O(5)–S(3)	1·409	O(6)–S(3)	1·377
F(7)–S(3)	1·562	O(8)–S(3)	1·337
O(9)–S(4)	1·395	O(10)–S(4)	1·431
F(11)–S(4)	1·520	O(12)–S(4)	1·308

Introduction. The structure determination was undertaken to establish the site of methylation of the heterocyclic ring. A preliminary note has been published (Crook, Jones, Kennard & Sykes, 1977). Large colourless crystals were obtained from ethanol. Intensities were determined with an automated Stoe two-circle diffractometer, Mo $K\alpha$ radiation with graphite monochromator, and a crystal 0·7 × 0·4 × 0·25 mm mounted about **b** (layers 0–16, 7234 reflexions). Lp corrections were applied; averaging equivalent reflexions then gave 5022 unique reflexions with $F > 4\sigma(F)$. Cell dimensions a, c and β were obtained by least-squares analysis of $h0l$ ω-angle measurements, and b from $0k0$ μ-angle measurements.

The structure was solved with *XCSD*; the best E map gave positions for all atoms of the two cations, and three positions (two of which were special positions $0,y,\frac{1}{4}$) were assigned to anion S atoms. The other anion atoms did not appear unambiguously. Anisotropic refinement of all located atoms, followed by a difference synthesis, showed the four missing atoms of the anion in the general position; a confusion of peaks near the other two S atoms was taken to indicate disorder. The anions were refined as follows: S(2) anion, all

Table 3. *Bond angles* (°)

C(5*A*)–S(1*A*)–N(2*A*)	95·2 (3)	C(5*B*)–S(1*B*)–N(2*B*)	95·3 (3)
N(3*A*)–N(2*A*)–S(1*A*)	110·4 (4)	N(3*B*)–N(2*B*)–S(1*B*)	109·3 (5)
C(4*A*)–N(3*A*)–N(2*A*)	115·5 (5)	C(4*B*)–N(3*B*)–N(2*B*)	117·7 (5)
C(6*A*)–N(3*A*)–N(2*A*)	118·3 (5)	C(6*B*)–N(3*B*)–N(2*B*)	117·5 (6)
C(6*A*)–N(3*A*)–C(4*A*)	126·1 (4)	C(6*B*) N(3*B*) -C(4*B*)	124·8 (5)
C(5*A*)–C(4*A*)–N(3*A*)	111·1 (4)	C(5*B*)–C(4*B*)–N(3*B*)	109·9 (5)
C(11*A*)–C(4*A*)–N(3*A*)	120·6 (5)	C(11*B*)–C(4*B*)–N(3*B*)	121·8 (5)
C(11*A*)–C(4*A*)–C(5*A*)	128·1 (5)	C(11*B*)–C(4*B*)–C(5*B*)	128·3 (5)
C(4*A*)–C(5*A*)–S(1*A*)	107·8 (4)	C(4*B*)–C(5*B*)–S(1*B*)	107·9 (4)
C(21*A*)–C(5*A*)–S(1*A*)	122·3 (4)	C(21*B*)–C(5*B*)–S(1*B*)	123·5 (4)
C(21*A*)–C(5*A*)–C(4*A*)	129·8 (4)	C(21*B*)–C(5*B*)–C(4*B*)	128·6 (5)
C(12*A*)–C(11*A*)–C(4*A*)	122·0 (4)	C(12*B*)–C(11*B*)–C(4*B*)	121·7 (5)
C(16*A*)–C(11*A*)–C(4*A*)	117·5 (4)	C(16*B*)–C(11*B*)–C(4*B*)	118·7 (4)
C(16*A*)–C(11*A*)–C(12*A*)	120·5 (4)	C(16*B*)–C(11*B*)–C(12*B*)	119·6 (5)
C(13*A*)–C(12*A*)–C(11*A*)	120·5 (5)	C(13*B*)–C(12*B*)–C(11*B*)	118·8 (6)
C(14*A*)–C(13*A*)–C(12*A*)	119·7 (6)	C(14*B*)–C(13*B*)–C(12*B*)	121·2 (6)
C(15*A*)–C(14*A*)–C(13*A*)	121·4 (6)	C(15*B*)–C(14*B*)–C(13*B*)	120·3 (7)
C(16*A*)–C(15*A*)–C(14*A*)	118·7 (5)	C(16*B*)–C(15*B*)–C(14*B*)	120·2 (6)
C(15*A*)–C(16*A*)–C(11*A*)	119·1 (5)	C(15*B*)–C(16*B*)–C(11*B*)	119·8 (5)
C(22*A*)–C(21*A*)–C(5*A*)	120·5 (5)	C(22*B*)–C(21*B*)–C(5*B*)	120·8 (5)
C(26*A*)–C(21*A*)–C(5*A*)	118·8 (5)	C(26*B*)–C(21*B*)–C(5*B*)	119·7 (5)
C(26*A*)–C(21*A*)–C(22*A*)	120·6 (6)	C(26*B*)–C(21*B*)–C(22*B*)	119·4 (6)
C(23*A*)–C(22*A*)–C(21*A*)	118·7 (6)	C(23*B*)–C(22*B*)–C(21*B*)	119·5 (5)
C(24*A*)–C(23*A*)–C(22*A*)	122·3 (7)	C(24*B*)–C(23*B*)–C(22*B*)	*120·4 (6)*
C(25*A*)–C(24*A*)–C(23*A*)	119·1 (7)	C(25*B*)–C(24*B*)–C(23*B*)	120·4 (7)
C(26*A*)–C(25*A*)–C(24*A*)	120·5 (6)	C(26*B*)–C(25*B*)–C(24*B*)	120·1 (6)
C(25*A*)–C(26*A*)–C(21*A*)	118·8 (6)	C(25*B*)–C(26*B*)–C(21*B*)	120·1 (5)
O(2)–S(2)–O(1)	114·5	O(3)–S(2)–O(1)	109·1
O(3)–S(2)–O(2)	109·0	O(4)–S(2)–O(1)	108·8
O(4)–S(2)–O(2)	110·6	O(4)–S(2)–O(3)	104·3
O(6)–S(3)–O(5)	112·4	F(7)–S(3)–O(5)	99·6
F(7)–S(3)–O(6)	105·5	O(8)–S(3)–O(5)	113·7
O(8)–S(3)–O(6)	119·8	O(8)–S(3)–F(7)	102·8
O(10)–S(4)–O(9)	107·3	F(11)–S(4)–O(9)	106·3
F(11)–S(4)–O(10)	99·1	O(12)–S(4)–O(9)	119·5
O(12)–S(4)–O(10)	114·4	O(12)–S(4)–F(11)	108·1

peripheral atoms assigned as O, since no bond-length difference was observed to distinguish S—F from S—O; this anion therefore exhibits O/F disorder: S(3) and S(4) anions, twofold disordered about the axis on which the S atoms lie; the O and F atoms could be distinguished by their bond lengths to S. In addition, the following distances were constrained to be equal within the given e.s.d.'s by the addition of extra observational equations to the least-squares matrix: all S—O (0·02 Å), both S—F (0·02 Å), all non-bonded O···F (0·04 Å), and all non-bonded O···O (0·04 Å). High resulting thermal parameters for O and F indicate, however, that the disorder may be more severe than that allowed for here.

In the final stages of refinement H atoms were included at calculated positions (C—H fixed at 1·08 Å) with an overall isotropic temperature factor. All refinement employed blocked full-matrix least squares with three blocks (cation 1, cation 2 and all anions respectively), interlayer scale factors being refined in each cycle. Convergence was achieved at $R = 0{\cdot}0831$ with a corresponding $R' = \Sigma w^{1/2}\Delta/\Sigma w^{1/2}|F_o|$ of 0·0893; the weighting scheme was $w = 1/[\sigma^2(F) + 0{\cdot}001F^2]$. Final values of the constrained distances were: S—O, 1·405 (6); S—F, 1·541 (15); F···O, 2·29 (2); O···O, 2·293 (11) Å. A final difference map had no peaks > 0·42 e Å^{-3}. Final positional parameters are given in Table 1, with derived bond lengths and angles in Tables 2 and 3.* The atomic numbering system is shown in Fig. 1.

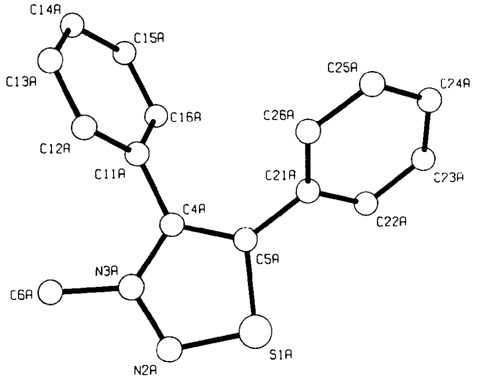

Fig. 1. Cation *A* showing the atomic numbering system. H atoms (omitted) are given the number of the C atom to which they are bonded. Cation *B* is numbered in the same way.

Discussion. The heterocyclic rings of the two independent cations show no significant differences in bond lengths and angles; the phenyl rings show some deviation from ideal geometry (bond lengths 1·339 to 1·397 Å). All rings are planar (maximum deviation from planarity 0·01 Å). Angles between mean ring planes are: heterocycle–phenyl 1: 118·5 and 104·3°; heterocycle–phenyl 2: 43·5 and 52·4° (cations *A* and *B* respectively). The substituent atoms on the heterocyclic rings are significantly out of the ring plane; deviations are C(6*A*) 0·02, C(11*A*) 0·10, C(21*A*) 0·06, C(6*B*) 0·02, C(11*B*) 0·05, C(21*B*) 0·05 Å. The angles C(6)–N(3)–C(4), C(11)–C(4)–C(5) and C(21)–C(5)–C(4) are considerably greater than 120° in both cations.

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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 33063 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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8-Iodoguanosine Monohydrate

By Jenan H. Al-Mukhtar and H. R. Wilson

*Carnegie Laboratory of Physics, University of Dundee, Dundee DD*1 *4HN, Scotland*

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Abstract. $C_{10}H_{12}N_5O_5I.H_2O$, monoclinic, $P2_1$, $a = 6{\cdot}981$ (5), $b = 11{\cdot}139$ (7), $c = 10{\cdot}196$ (7) Å, $\beta = 108{\cdot}1$ (1)°, $U = 753{\cdot}7$ Å^3, $Z = 2$, $D_x = 1{\cdot}886$ g cm^{-3}. Linear diffractometer data using Mo $K\alpha$ radiation. The structure was refined to an R of 0·085 for 1296 unique reflexions. The molecule is in the *syn* confor-