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Structure and Conformation of the 1:1 Molecular Complex Sulfaproxyline–Caffeine

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Abstract. *p*-Isopropoxy-*N*-sulfanilylbenzamide–3,7-dihydro-1,3,7-trimethyl-1*H*-purine-2,6-dione (1/1), $C_{16}H_{18}N_2O_4S.C_8H_{10}N_4O_2$, $M_r = 528{\cdot}58$, monoclinic, $P2_1/c$, $Z = 8$, $a = 12{\cdot}197$ (1), $b = 34{\cdot}109$ (5), $c = 12{\cdot}878$ (1) Å, $\beta = 108{\cdot}68$ (1)°, $V = 5075{\cdot}4$ (1·0) Å^3, $D_x = 1{\cdot}38$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}5418$ Å, $\mu = 15{\cdot}32$ cm^{-1}, $F(000) = 2224$, $T = 293$ K, final $R = 0{\cdot}046$ for 5997 observed reflections with $F \geq 3\sigma(F)$. The asymmetric unit contains two sulfaproxyline and two caffeine molecules. In the crystal the flat caffeine molecule is packed parallel to another caffeine molecule. Hydrogen bonds, where present, are weak ($D\cdots A$ lengths > 3·0 Å), showing that the packing is dominated by the non-bonded interactions.

Introduction. Sulfonamides form molecular complexes with different small molecules. Trimethoprim–sulfamethoxazole (Nakai, Takasuka & Shiro, 1984), sulfathiazole–xanthine (Higuchi & Lach, 1954), sulfathiazole–theophylline (Shefter & Sackman, 1971) *etc.* are such molecular complexes. The molecular basis for complex formation in such a simple system helps in the understanding of the interaction of small molecules with a bioreceptor and of the resulting antibacterial activity.

Here crystallographic studies on such a sulfonamide complex (1:1 sulfaproxyline–caffeine) have been carried out to determine the nature of the interaction between the compounds, hydrogen bonding and change in structural features, if any, due to this complex formation. Sulfaproxyline is widely used in synergestic combination with sulfamerazine, as the metabolites are readily soluble in acid urine preventing the formation of crystalluria even at acidic pH. Caffeine, a xanthine derivative, stimulates the central nervous system, cardiac muscle, cortex and respiratory system.

Experimental. Needle-shaped light-yellowish crystals were obtained by slow evaporation of an acetone solution of a 1:1 mixture of sulfaproxyline and caffeine. Weissenberg photographs showed the crystals to be monoclinic and systematic absence conditions determined the space group as $P2_1/c$. The lattice parameters a, b, c and β were also found using oscillation as well as Weissenberg photographs.

A crystal of dimensions 0·26 × 0·08 × 0·05 mm was used for intensity data collection on an Enraf–Nonius CAD-4F diffractometer using Cu $K\alpha$ radiation. The cell parameters were determined by least-squares refinement of 48 reflections with $19 < \theta < 42°$. ω–2θ scans. The data were measured for values of 2θ up to 120° with $-13 \leq h \leq 13$, $0 \leq k \leq 38$ and $-14 \leq l \leq 14$. The reflection $\bar{3}57$ was measured at regular intervals as an intensity check with an average count of 449·5 and σ (calculated from distribution) = 7·2 (1·6%). The data were corrected for Lorentz and polarization factors but not for absorption. 11 447 values were recorded and merged using *SHELX76* (Sheldrick, 1976) to give 6454 unique reflections (merging $R = 0{\cdot}02$). The structure was solved by direct methods using *MULTAN78* (Main,

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Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined isotropically by full-matrix least squares based on F using *SHELX*76 with 5997 observed [$F \geq 3\sigma(F)$] reflections. Owing to the limitation of the total number of parameters in *SHELX*76 each pair of molecules forming half of the asymmetric unit was refined anisotropically in turn. H atoms were located from a difference Fourier synthesis and put into the atom list with the equivalent isotropic thermal parameter of the attached atom.

Further refinement gave $R = 0{\cdot}046$, $wR = 0{\cdot}051$ with $w = 1{\cdot}7237/(\sigma^2 F_o + 0{\cdot}001297|F_o|^2)$. In the final refinement cycle, the average shift to e.s.d. ratio was 0·1; maximum and minimum electron densities in the final ΔF map were 0·295 and $-0{\cdot}290$ e Å^{-3}, respectively. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Discussion. The final atomic parameters are listed in Table 1* and bond distances, angles and selected torsion angles are given in Table 2. Two complex molecules are present in each asymmetric unit. Fig. 1 shows the molecular diagram with the atomic numbering scheme.

Sulfaproxyline possesses various conformations about the C(1)—S, S—N(1) and N(1)—C(7) bonds. The torsion angles $|\varepsilon_1|$ [C(X)—C(1)—S—N(1), where $X = 2$ or 6] and $|\varepsilon_2|$ [C(1)—S—N(1)—C(7)], defining the conformation of the sulfonamide group, lie in the range 70–120 and 60–90°, respectively, found in related molecules (Kálmán, Czugler & Argay, 1981). But the values are different in the free sulfaproxyline molecule (Basak, Mazumdar & Chaudhuri, 1984) where $\varepsilon_1 = -84{\cdot}1$ (3), 103·8 (3)° and $\varepsilon_2 = 75{\cdot}6$ (2)°. The torsion angles S—N(1)—C(7)—C(8) for molecules A and B of present structure are greater than the value of 161·4 (2)° in the free molecule. The conformations of sulfaproxyline in the free molecule and in the present structure about the S—N(1) bond are shown in Fig. 2, in which Newman projections along the same bond are compared.

The two planar rings are almost perpendicular to each other, the dihedral angle between them being 82·9 (1) and 82·0 (1)° for molecules A and B, respectively. However, this dihedral angle in the free molecule is 51·9 (1)°, which is significantly smaller than those in the present structure and the values observed in most of the sulfonamides.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, bond lengths and angles involving H atoms, and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53270 (42 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and U_{eq} values (Å^2) for non-*H *atoms with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
Molecule *A*				
S	0·2404 (1)	0·3129 (0)	0·5784 (1)	0·0412 (2)
N(1)	0·3147 (2)	0·3089 (1)	0·7116 (2)	0·0417 (8)
N(2)	0·0056 (2)	0·3557 (1)	0·2801 (2)	0·0522 (9)
N(3)	0·1536 (2)	0·3355 (1)	0·2095 (2)	0·0635 (10)
N(4)	0·3844 (2)	0·4633 (1)	0·4378 (2)	0·0603 (10)
N(5)	0·1213 (2)	0·4430 (1)	0·2170 (2)	0·0636 (10)
N(6)	−0·0172 (2)	0·4256 (1)	0·2916 (2)	0·0500 (9)
O(1)	0·2729 (2)	0·2813 (1)	0·5223 (1)	0·0534 (8)
O(2)	0·1228 (1)	0·3159 (1)	0·5773 (2)	0·0513 (8)
O(3)	0·4921 (1)	0·3046 (1)	0·6896 (1)	0·0500 (8)
O(4)	0·6527 (2)	0·2939 (1)	1·2074 (1)	0·0549 (7)
O(5)	0·2614 (2)	0·3800 (1)	0·1491 (2)	0·0881 (13)
O(6)	0·0393 (2)	0·2910 (1)	0·2599 (2)	0·0878 (12)
C(1)	0·2812 (2)	0·3572 (1)	0·5342 (2)	0·0401 (9)
C(2)	0·2210 (2)	0·3915 (1)	0·5375 (2)	0·0430 (9)
C(3)	0·2541 (2)	0·4265 (1)	0·5056 (2)	0·0470 (11)
C(4)	0·3500 (2)	0·4286 (1)	0·4687 (2)	0·0442 (10)
C(5)	0·4095 (2)	0·3941 (1)	0·4638 (2)	0·0505 (11)
C(6)	0·3760 (2)	0·3589 (1)	0·4964 (2)	0·0463 (10)
C(7)	0·4336 (2)	0·3048 (1)	0·7512 (2)	0·0396 (9)
C(8)	0·4866 (2)	0·3013 (1)	0·8719 (2)	0·0399 (9)
C(9)	0·4295 (2)	0·2846 (1)	0·9388 (2)	0·0489 (10)
C(10)	0·4818 (2)	0·2817 (1)	1·0501 (2)	0·0525 (11)
C(11)	0·5929 (2)	0·2961 (1)	1·0975 (2)	0·0443 (9)
C(12)	0·6528 (2)	0·3117 (1)	1·0319 (2)	0·0490 (11)
C(13)	0·6000 (2)	0·3140 (1)	0·9204 (2)	0·0457 (10)
C(14)	0·5889 (2)	0·2978 (1)	1·2842 (2)	0·0526 (11)
C(15)	0·6716 (3)	0·2872 (1)	1·3939 (3)	0·0786 (15)
C(16)	0·5437 (4)	0·3389 (1)	1·2790 (4)	0·1052 (19)
C(17)	0·0646 (3)	0·3251 (1)	0·2505 (2)	0·0629 (14)
C(18)	0·1848 (3)	0·3739 (1)	0·1888 (2)	0·0597 (13)
C(19)	0·1172 (2)	0·4026 (1)	0·2196 (2)	0·0487 (10)
C(20)	0·0330 (2)	0·3939 (1)	0·2653 (2)	0·0460 (10)
C(21)	0·0392 (3)	0·4555 (1)	0·2613 (3)	0·0658 (14)
C(22)	0·2205 (3)	0·3028 (1)	0·1863 (4)	0·0957 (19)
C(23)	−0·0907 (3)	0·3463 (1)	0·3191 (3)	0·0735 (14)
C(24)	0·1941 (3)	0·4677 (1)	0·1750 (3)	0·0852 (17)
Molecule *B*				
S	0·8758 (1)	0·0558 (0)	1·0618 (1)	0·0459 (3)
N(1)	0·8222 (2)	0·0538 (1)	0·9261 (2)	0·0460 (8)
N(2)	1·1177 (2)	0·1081 (1)	1·3663 (2)	0·0435 (8)
N(3)	0·9633 (2)	0·0973 (1)	1·4358 (2)	0·0490 (9)
N(4)	0·7476 (2)	0·2096 (1)	1·1947 (2)	0·0693 (11)
N(5)	1·0295 (2)	0·2019 (1)	1·4166 (2)	0·0527 (9)
N(6)	1·1630 (2)	0·1765 (1)	1·3485 (2)	0·0496 (9)
O(1)	0·8191 (2)	0·0258 (1)	1·1029 (2)	0·0628 (8)
O(2)	0·9983 (2)	0·0540 (1)	1·0836 (2)	0·0592 (8)
O(3)	0·6320 (2)	0·0541 (1)	0·9126 (2)	0·0607 (7)
O(4)	0·6160 (2)	0·0301 (1)	0·4152 (1)	0·0512 (7)
O(5)	0·8714 (2)	0·1477 (1)	1·4917 (2)	0·0777 (10)
O(6)	1·0559 (2)	0·0462 (1)	1·3843 (2)	0·0663 (9)
C(1)	0·8384 (2)	0·1013 (1)	1·1016 (2)	0·0407 (10)
C(2)	0·7351 (2)	0·1060 (1)	1·1239 (2)	0·0515 (10)
C(3)	0·7058 (2)	0·1417 (1)	1·1543 (2)	0·0541 (11)
C(4)	0·7780 (2)	0·1740 (1)	1·1635 (2)	0·0475 (9)
C(5)	0·8821 (2)	0·1690 (1)	1·1416 (2)	0·0493 (11)
C(6)	0·9113 (2)	0·1331 (1)	1·1108 (2)	0·0444 (10)
C(7)	0·7059 (2)	0·0511 (1)	0·8679 (2)	0·0465 (9)
C(8)	0·6792 (2)	0·0444 (1)	0·7489 (2)	0·0446 (10)
C(9)	0·5778 (2)	0·0593 (1)	0·6761 (2)	0·0511 (01)
C(10)	0·5519 (2)	0·0550 (1)	0·5645 (2)	0·0507 (10)
C(11)	0·6279 (2)	0·0351 (1)	0·5231 (2)	0·0435 (9)
C(12)	0·7263 (2)	0·0176 (1)	0·5953 (2)	0·0433 (10)
C(13)	0·7517 (2)	0·0227 (1)	0·7063 (2)	0·0434 (9)
C(14)	0·5185 (2)	0·0475 (1)	0·3314 (2)	0·0505 (10)
C(15)	0·5302 (3)	0·0911 (1)	0·3272 (3)	0·0784 (16)
C(16)	0·5212 (3)	0·0278 (1)	0·2268 (2)	0·0670 (13)
C(17)	1·0462 (2)	0·0817 (1)	1·3946 (2)	0·0481 (11)
C(18)	0·9453 (2)	0·1369 (1)	1·4525 (2)	0·0489 (10)
C(19)	1·0220 (2)	0·1616 (1)	1·4195 (2)	0·0421 (10)
C(20)	1·1037 (2)	0·1476 (1)	1·3775 (2)	0·0400 (9)
C(21)	1·1144 (3)	0·2091 (1)	1·3733 (3)	0·0583 (12)
C(22)	0·8868 (3)	0·0688 (1)	1·4646 (3)	0·0723 (16)
C(23)	1·2113 (3)	0·0929 (1)	1·3301 (3)	0·0698 (15)
C(24)	0·9623 (3)	0·2314 (1)	1·4534 (3)	0·0703 (14)

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

	Molecule *A*	Molecule *B*
Distances in sulfaproxyline		
S—N(1)	1·669 (2)	1·660 (3)
S—O(1)	1·423 (3)	1·428 (3)
N(1)—C(7)	1·382 (3)	1·379 (3)
C(7)—O(3)	1·225 (3)	1·219 (4)
C(14)—O(4)	1·447 (4)	1·452 (3)
C(14)—C(16)	1·500 (5)	1·515 (4)
C(1)—C(2)	1·389 (4)	1·388 (4)
C(3)—C(4)	1·398 (4)	1·392 (4)
C(5)—C(6)	1·376 (5)	1·369 (5)
C(8)—C(9)	1·391 (4)	1·387 (3)
C(10)—C(11)	1·386 (3)	1·386 (4)
C(12)—C(13)	1·375 (3)	1·373 (4)
S—C(1)	1·742 (3)	1·740 (3)
S—O(2)	1·433 (2)	1·430 (3)
N(4)—C(4)	1·357 (5)	1·367 (5)
C(11)—O(4)	1·371 (3)	1·361 (3)
C(14)—C(15)	1·494 (4)	1·497 (5)
C(7)—C(8)	1·485 (3)	1·479 (4)
C(2)—C(3)	1·365 (5)	1·361 (5)
C(4)—C(5)	1·394 (4)	1·396 (4)
C(6)—C(1)	1·392 (4)	1·383 (4)
C(9)—C(10)	1·373 (3)	1·377 (4)
C(11)—C(12)	1·388 (4)	1·395 (3)
C(13)—C(8)	1·392 (3)	1·393 (4)
Distances in caffeine		
N(3)—C(17)	1·396 (5)	1·389 (4)
N(3)—C(22)	1·468 (5)	1·475 (5)
N(2)—C(20)	1·373 (5)	1·371 (5)
N(5)—C(19)	1·380 (5)	1·379 (5)
N(5)—C(24)	1·449 (5)	1·469 (5)
N(6)—C(21)	1·356 (5)	1·346 (5)
C(17)—O(6)	1·219 (5)	1·228 (5)
N(3)—C(18)	1·413 (5)	1·396 (5)
N(2)—C(17)	1·389 (5)	1·381 (4)
N(2)—C(23)	1·453 (5)	1·460 (5)
N(5)—C(21)	1·370 (5)	1·347 (5)
N(6)—C(20)	1·338 (5)	1·344 (4)
C(18)—O(5)	1·218 (5)	1·222 (4)
C(19)—C(20)	1·370 (4)	1·363 (4)
C(18)—C(19)	1·416 (5)	1·421 (4)
Bond angles in sulfaproxyline		
O(1)—S—C(1)	109·6 (1)	109·0 (2)
O(1)—S—O(2)	119·3 (1)	119·7 (2)
O(1)—S—N(1)	108·2 (2)	106·6 (2)
S—N(1)—C(7)	123·2 (2)	124·4 (2)
O(3)—C(7)—C(8)	121·9 (2)	123·5 (3)
N(4)—C(4)—C(3)	121·0 (3)	120·3 (3)
C(2)—C(1)—C(6)	118·8 (3)	119·5 (3)
C(2)—C(3)—C(4)	120·5 (3)	121·0 (3)
C(4)—C(5)—C(6)	120·8 (3)	120·4 (3)
S—C(1)—C(2)	120·7 (2)	120·3 (3)
C(7)—C(8)—C(9)	123·0 (2)	119·8 (2)
C(9)—C(8)—C(13)	118·2 (2)	118·0 (2)
C(9)—C(10)—C(11)	119·7 (2)	119·7 (2)
C(11)—C(12)—C(13)	119·7 (3)	119·9 (3)
O(4)—C(11)—C(10)	123·9 (2)	126·0 (2)
O(4)—C(14)—C(15)	106·0 (2)	111·6 (2)
C(11)—O(4)—C(14)	118·4 (2)	120·1 (2)
O(2)—S—C(1)	108·9 (2)	109·6 (2)
C(1)—S—N(1)	106·8 (2)	107·1 (2)
O(2)—S—N(1)	103·2 (1)	103·8 (2)
O(3)—C(7)—N(1)	121·4 (2)	121·7 (2)
N(1)—C(7)—C(8)	116·6 (2)	114·9 (2)
N(4)—C(4)—C(5)	120·5 (3)	121·2 (3)
C(1)—C(2)—C(3)	121·1 (3)	120·1 (3)
C(3)—C(4)—C(5)	118·4 (3)	118·5 (3)
C(5)—C(6)—C(1)	120·3 (3)	120·4 (3)
S—C(1)—C(6)	120·4 (3)	120·2 (2)
C(7)—C(8)—C(13)	118·8 (2)	122·2 (2)
C(8)—C(9)—C(10)	121·2 (3)	121·5 (3)
C(10)—C(11)—C(12)	119·9 (2)	119·5 (2)
C(8)—C(13)—C(12)	121·1 (3)	121·1 (3)
O(4)—C(11)—C(12)	116·1 (2)	114·5 (2)
O(4)—C(14)—C(16)	109·0 (2)	103·5 (2)
O(15)—C(14)—C(16)	113·3 (3)	112·3 (3)
Bond angles in caffeine		
C(17)—N(3)—C(18)	126·6 (3)	126·8 (3)
C(18)—N(3)—C(22)	117·7 (3)	117·1 (3)
C(17)—N(2)—C(23)	118·4 (3)	118·5 (3)
C(19)—N(5)—C(21)	105·3 (3)	105·0 (3)

Table 2 (*cont.*)

	Molecule *A*	Molecule *B*
C(21)—N(5)—C(24)	126·3 (3)	126·2 (3)
N(5)—C(21)—N(6)	113·1 (3)	113·8 (3)
O(6)—C(17)—N(2)	121·3 (3)	121·6 (3)
O(5)—C(18)—C(19)	126·4 (3)	126·0 (3)
N(6)—C(20)—C(19)	113·6 (3)	112·3 (3)
C(18)—C(19)—C(20)	123·7 (3)	123·1 (3)
N(2)—C(20)—N(6)	125·4 (3)	126·5 (3)
C(20)—C(19)—N(5)	105·3 (3)	106·0 (3)
C(17)—N(3)—C(22)	115·7 (3)	116·1 (3)
C(17)—N(2)—C(20)	120·3 (3)	120·1 (3)
C(20)—N(2)—C(23)	121·1 (3)	121·3 (3)
C(19)—N(5)—C(24)	128·4 (3)	128·7 (3)
C(20)—N(6)—C(21)	102·7 (3)	102·9 (3)
O(6)—C(17)—N(3)	122·1 (3)	121·7 (3)
O(5)—C(18)—N(3)	121·8 (3)	121·9 (3)
N(2)—C(17)—N(3)	116·5 (3)	116·7 (3)
N(3)—C(18)—C(19)	111·8 (3)	112·1 (3)
C(19)—C(20)—N(2)	120·9 (3)	121·1 (3)
C(18)—C(19)—N(5)	130·9 (3)	130·9 (3)
Torsion angles in sulfaproxyline		
C(2)—C(1)—S—N(1)	−90·6 (3)	89·7 (3)
C(2)—C(1)—S—O(1)	152·5 (2)	−25·3 (3)
C(2)—C(1)—S—O(2)	20·2 (3)	−158·2 (2)
C(1)—S—N(1)—C(7)	−66·0 (3)	−70·1 (3)
O(2)—S—N(1)—C(7)	179·2 (3)	173·8 (3)
S—N(1)—C(7)—O(3)	1·4 (4)	7·8 (5)
C(6)—C(1)—S—N(1)	88·5 (3)	−90·0 (3)
C(6)—C(1)—S—O(1)	−28·5 (3)	154·9 (3)
C(6)—C(1)—S—O(2)	−160·7 (2)	22·1 (3)
O(1)—S—N(1)—C(7)	51·9 (3)	46·5 (3)
S—N(1)—C(7)—C(8)	−179·5 (2)	−172·2 (2)

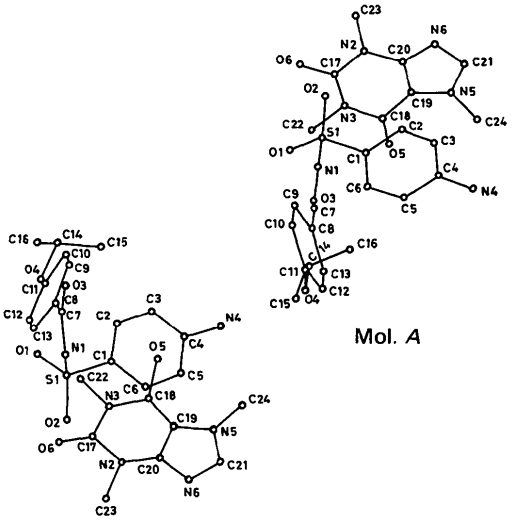

Fig. 1. Molecular diagram.

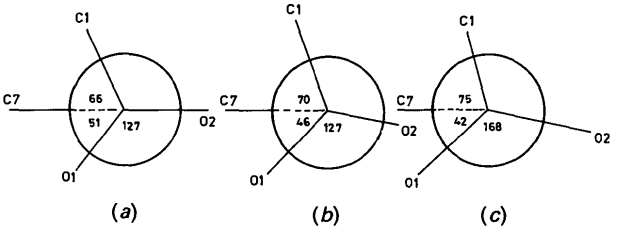

Fig. 2. Newman projection showing characteristic features of sulfaproxyline along the S—N(1) bond. (*a*) Molecule *A* and (*b*) molecule *B* in the present structure. (*c*) Free molecule.

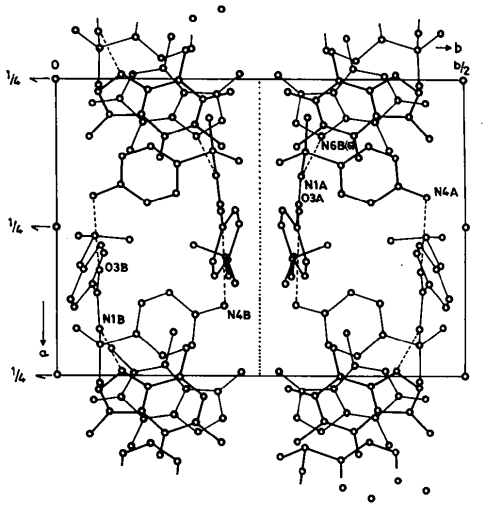

Fig. 3. Molecular packing diagram viewed along the *c* axis.

The pyrimidine and imidazole rings of the caffeine molecule are planar and the dihedral angle between them is 1·6 (1) and 2·6 (1)° for molecules *A* and *B*, respectively; the dihedral angle between them is 67·0 (2)° in the free molecule (Sutor, 1958).

Intermolecular bonds and molecular packing. Molecular packing in the crystal is shown in Fig. 3. Three hydrogens are available for hydrogen bonding in each molecule, two from the aryl amino nitrogen and one from the sulfonamido nitrogen. But H(42)*A* and H(41)*B* do not participate in hydrogen bonding.

Table 3. *Hydrogen-bond geometry* (Å, °)

D—H···*A*	*D*—H	*D*···*A*	H···*A*	*D*—H···*A*
N(4)*B*—H(42)*B*···O(3^{i})*A*	0·79 (3)	3·134 (3)	2·38 (3)	160·7 (2·6)
N(4)*A*—H(41)*A*···O(3^{ii})*B*	0·79 (3)	3·195 (4)	2·45 (3)	159·3 (2·6)
N(1)*B*—H(1)*B*···N(6^{iii})*A*	0·80 (3)	3·083 (3)	2·29 (3)	171·4 (2·3)
N(1)*A*—H(1)*A*···N(6^{iv})*B*	0·81 (3)	2·978 (4)	2·17 (3)	172·0 (2·1)

Symmetry code: (i) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (ii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) x, y, z; (iv) $x-1, -y+\frac{1}{2}, z-\frac{1}{2}$.

The hydrogen-bonding geometry (Table 3) shows weak hydrogen bonds having $D\cdots A$ lengths > 3·0 Å. The predominant intermolecular forces responsible for complex formation are therefore non-bonded interactions. The flat caffeine molecules are packed parallel to each other.

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Structure of an Adenine Tetrafluoroborate Salt

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Abstract. Adenine–adeninium tetrafluoroborate–water (1/1/2), $C_5H_5N_5.C_5H_6N_5^+.BF_4^-.2H_2O$, M_r = 394·10, monoclinic, $P2_1/m$, a = 5·358 (1), b = 19·468 (7), c = 7·905 (2) Å, β = 104·75 (2)°, V = 797·4 (7) Å^3, Z = 2, D_x = 1·641 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 1·42 cm^{-1}, $F(000)$ = 404, T = 298 K. Final R = 0·062 for 1597 unique observed reflections with $I_o > 2\sigma(I_o)$. After consideration of various factors the structure was assigned as adenine and adeninium moieties in a 1:1 ratio, disordered over the one site. The BF_4^- anion and the two water molecules of crystallization lie on mirror planes. H atoms on C(2), N(6), C(8) and N(9) of adenine were located in difference-Fourier maps and were refined, but those

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