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Structure of Arcaine Sulphate, $C_6H_{18}N_6^{2+}.SO_4^{2-}$

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Abstract. $M_r = 270{\cdot}3$, monoclinic, $P2_1/n$, $a = 7{\cdot}263$ (2), $b = 35{\cdot}140$ (9), $c = 15{\cdot}424$ (4) Å, $\beta = 101{\cdot}70$ (3)°, $V = 3854{\cdot}8$ Å^3, $Z = 12$, $D_x = 1{\cdot}397$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 2{\cdot}29$ mm^{-1}, $T = 296$ K, $F(000) = 1728$, final $R(F) = 0{\cdot}104$ for 2649 reflections. There are three amine sulphate molecules per asymmetric unit leading to interesting amine–sulphate interactions through an extensive N—H···O-type hydrogen-bond network. The molecules are in the all-*trans* configuration, except for one of the two guanidyl groups in each molecule.

Introduction. Polyamines are low-molecular-weight aliphatic, nitrogenous bases. Polyamines produced from amino acids by bacteria have pharmacological activities in animals. Studies on polyamines have thrown light on the mode of binding of polyamines to DNA (Pattabhi & Chandrasekhar, 1983; Liquori *et al.*, 1967; Woo, Seeman & Rich, 1979; Tsuboi, 1964).

In view of their importance in biological processes, the crystal structure analysis of arcaine sulphate was undertaken as part of the project on studies of polyamines and their interactions.

Experimental. Crystal 0·3 × 0·2 × 0·2 mm, $\theta/2\theta$ scan with line profile analysis (Grant & Gabe, 1978); Picker four-circle automatic diffractometer, graphite-monochromatized Cu $K\alpha$; 5720 independent reflections with $\theta < 60°$ giving the range of h, k and l as −8 to 7, 0 to 39 and 0 to 17 respectively; 2649 with $I_{net} > 2\sigma(I_{net})$; three standard reflections measured after every 100 regular reflections, no significant fluctuations observed; data corrected for direct-beam polarization (Le Page, Gabe & Calvert, 1979) and Lorentz effects; no absorption correction; unit-cell parameters determined from least-squares refinement of angle values for 42 reflections with $30 < \theta < 40°$; structure solution by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); anisotropic full-matrix refinement using F_o; 45 out of 54 H atoms (from ΔF synthesis and geometry) included only in structure factor calculations; final $R(F) = 0{\cdot}104$, $R_w(F) = 0{\cdot}062$; $w = 1/\sigma^2(F_o)$ based on counting statistics; goodness of fit = 3·86; R(all) = 0·204, R_w(all) = 0·064; final difference map had no peaks > 0·58 e Å^{-3}; $(\Delta/\sigma)_{max} = 1{\cdot}0$, $(\Delta/\sigma)_{mean} = 0{\cdot}2$. Repeated attempts at crystallization under various conditions failed and the determination was carried out with the available crystals, though the poor quality of these crystals restricted the accuracy of the structure. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations performed using the NRC-PDP-8e system of programs (Larson & Gabe, 1978) adapted for the VAX computer.

Discussion. Atomic positions and equivalent isotropic temperature factors for non-hydrogen atoms are listed in Table 1.‡ A stereoview of the molecule is shown in Fig. 1. Interatomic distances and angles are given in

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‡ Lists of structure factors, anisotropic thermal parameters and torsion angles and details of the hydrogen-bond geometry have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39723 (47 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 2. The coordination of the sulphate ions is tetrahedral. It has been observed in earlier studies that the amine molecules exist in both *trans* and *gauche* conformations, though the all-*trans* conformation is more prevalent. The conformational preference seems to be controlled by the packing forces. The atoms N(16) through N(24) of molecule (I), N(29) through N(36) of molecule (II) and N(41) through N(48) of molecule (III) are in the all-*trans* configuration with the following average bond lengths and angles in the butylamine segment: C–C 1·51 (2), C–N 1·48 (2) Å, ∠C–C–C 112 (2), ∠N–C–C 113 (2)°. These values are within 3σ of values tabulated earlier (Chandrasekhar & Pattabhi, 1980; Chandrasekhar, Pattabhi & Raghunathan, 1982). The torsion angles around C(23)–N(24) of molecule (I), C(35)–N(36) of molecule (II), C(47)–N(48) of molecule (III) are 93, −85 and 80°, respectively. The guanidyl groups are triangular planar ($\chi^2 < 1{\cdot}02$) and the mean C–N, C=N lengths are 1·37 (2), 1·31 (2) Å and the N–C–N angle is 120 (2)°.

The molecule exhibits a layered packing with the zigzag amine chains arranged in planes parallel to *bc*. The three butylamine segments are approximately in parallel planes, the angles between these planes being 168 (2), 12 (2), 167 (2)°. The sulphate ions interleave these planes as observed in other amine sulphate and phosphate structures (Chandrasekhar *et al.*, 1982; Huse & Iitaka, 1969).

There are eight protons from the amines and two from the SO_4^{2-} groups for hydrogen bonding per

Table 1. *Fractional positional parameters and* B_{eq} *temperature factors* (Å²) *for non-hydrogen atoms*

B_{eq} is the mean of the principal axes of the thermal ellipsoid.

	x	*y*	*z*	B_{eq}
S(1)	0·6216 (6)	0·41634 (14)	0·2066 (3)	2·80 (3)
S(2)	0·4539 (6)	0·58222 (15)	0·2954 (3)	2·462 (18)
S(3)	0·3783 (7)	0·25145 (14)	0·8038 (3)	2·52 (4)
O(4)	0·6178 (16)	0·3746 (3)	0·1958 (8)	4·3 (7)
O(5)	0·6889 (16)	0·4260 (4)	0·3040 (8)	4·8 (7)
O(6)	0·7605 (15)	0·4332 (3)	0·1569 (7)	3·7 (6)
O(7)	0·4262 (15)	0·4312 (3)	0·1685 (8)	4·5 (7)
O(8)	0·4627 (15)	0·5403 (4)	0·3123 (8)	4·6 (8)
O(9)	0·3275 (16)	0·6012 (3)	0·3470 (8)	4·1 (7)
O(10)	0·6459 (15)	0·5971 (3)	0·3230 (8)	4·0 (6)
O(11)	0·3762 (15)	0·5893 (4)	0·1988 (7)	4·4 (7)
O(12)	0·3854 (15)	0·2930 (3)	0·8164 (9)	4·5 (8)
O(13)	0·2053 (15)	0·2360 (3)	0·8315 (8)	4·5 (7)
O(14)	0·5482 (14)	0·2335 (3)	0·8564 (8)	3·8 (6)
O(15)	0·3620 (15)	0·2441 (4)	0·7083 (6)	3·8 (7)
N(16)	0·3061 (19)	0·5589 (4)	1·0279 (10)	3·5 (8)
N(17)	0·3282 (19)	0·5068 (4)	1·1201 (9)	3·4 (8)
C(18)	0·2926 (24)	0·5197 (6)	1·0380 (13)	3·2 (11)
N(19)	0·2465 (19)	0·4983 (4)	0·9681 (9)	3·1 (8)
C(20)	0·2194 (24)	0·4565 (6)	0·9786 (12)	3·43 (11)
C(21)	0·1582 (25)	0·4384 (6)	0·8887 (13)	4·02 (10)
C(22)	0·131 (3)	0·3962 (6)	0·9044 (11)	3·56 (7)
C(23)	0·0715 (24)	0·3738 (7)	0·8204 (12)	4·70 (17)
N(24)	0·0509 (19)	0·3320 (4)	0·8369 (10)	3·1 (8)
C(25)	−0·1101 (24)	0·3140 (6)	0·8445 (12)	4·0 (11)
N(26)	−0·2728 (18)	0·3348 (4)	0·8393 (10)	3·6 (9)
N(27)	−0·1068 (20)	0·2760 (4)	0·8552 (10)	3·6 (8)
N(28)	0·2879 (19)	0·3249 (4)	0·6271 (10)	3·6 (8)
N(29)	0·1978 (19)	0·2708 (4)	0·5404 (10)	3·6 (8)
C(30)	0·2191 (22)	0·3092 (6)	0·5449 (10)	2·7 (9)
N(31)	0·1729 (18)	0·3320 (4)	0·4766 (9)	2·9 (7)
C(32)	0·1882 (23)	0·3744 (5)	0·4857 (11)	3·12 (8)
C(33)	0·1571 (22)	0·3935 (5)	0·3971 (13)	3·43 (14)
C(34)	0·158 (3)	0·4350 (5)	0·4083 (14)	4·16 (17)
C(35)	0·1439 (23)	0·4580 (5)	0·3232 (11)	3·17 (17)
N(36)	0·1324 (19)	0·4994 (4)	0·3363 (10)	3·3 (8)
C(37)	−0·029 (3)	0·5186 (5)	0·3378 (12)	3·2 (10)
N(38)	−0·0087 (20)	0·5574 (4)	0·3514 (9)	3·7 (8)
N(39)	−0·1950 (18)	0·5015 (4)	0·3330 (9)	3·8 (8)
N(40)	0·1770 (18)	0·1601 (4)	−0·1203 (9)	3·1 (7)
N(41)	0·1678 (18)	0·1062 (4)	−0·0339 (9)	3·0 (7)
C(42)	0·1867 (24)	0·1449 (5)	−0·0407 (13)	3·2 (10)
N(43)	0·2035 (20)	0·1654 (4)	0·0313 (10)	3·2 (8)
C(44)	0·2030 (22)	0·2064 (6)	0·0249 (10)	3·56 (6)
C(45)	0·2525 (23)	0·2266 (5)	0·1117 (12)	3·08 (14)
C(46)	0·2389 (25)	0·2691 (6)	0·1007 (12)	3·96 (18)
C(47)	0·2980 (24)	0·2935 (6)	0·1860 (12)	3·99 (14)
N(48)	0·2648 (20)	0·3341 (4)	0·1715 (10)	3·4 (8)
C(49)	0·1013 (24)	0·3542 (7)	0·1649 (12)	4·0 (13)
N(50)	0·0990 (22)	0·3909 (4)	0·1548 (11)	4·3 (9)
N(51)	−0·0521 (18)	0·3333 (4)	0·1704 (10)	4·0 (9)

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

The atom numbering for molecule (II) is the same as for molecule (I), plus 1 for S, 4 for O and 12 for C and N. Molecule (III) is similarly related to molecule (II).

	Molecule (I)	Molecule (II)	Molecule (III)
S(1)–O(4)	1·48 (1)	1·49 (1)	1·47 (1)
S(1)–O(5)	1·52 (1)	1·49 (1)	1·51 (1)
S(1)–O(6)	1·51 (1)	1·47 (1)	1·47 (1)
S(1)–O(7)	1·51 (1)	1·50 (1)	1·48 (1)
C(18)–N(16)	1·39 (3)	1·38 (2)	1·33 (2)
C(18)–N(17)	1·32 (2)	1·36 (2)	1·37 (2)
C(18)–N(19)	1·30 (2)	1·31 (2)	1·31 (2)
N(19)–C(20)	1·49 (2)	1·50 (2)	1·44 (2)
C(20)–C(21)	1·51 (3)	1·50 (2)	1·49 (2)
C(21)–C(22)	1·52 (3)	1·47 (3)	1·50 (3)
C(22)–C(23)	1·50 (2)	1·53 (3)	1·56 (3)
C(23)–N(24)	1·50 (3)	1·47 (2)	1·45 (2)
N(24)–C(25)	1·36 (2)	1·36 (2)	1·37 (2)
C(25)–N(26)	1·38 (2)	1·38 (2)	1·30 (3)
C(25)–N(27)	1·34 (2)	1·33 (2)	1·35 (2)
O(4)–S(1)–O(5)	109·1 (7)	111·0 (7)	109·2 (7)
O(5)–S(1)–O(6)	107·5 (7)	109·6 (7)	110·0 (7)
O(6)–S(1)–O(7)	109·7 (7)	111·8 (7)	111·4 (7)
O(7)–S(1)–O(5)	113·2 (7)	107·9 (7)	108·4 (7)
O(6)–S(1)–O(4)	109·5 (7)	107·2 (7)	110·3 (7)
O(7)–S(1)–O(4)	107·8 (7)	109·3 (7)	107·4 (8)
N(16)–C(18)–N(17)	116 (2)	117 (1)	119 (2)
N(16)–C(18)–N(19)	119 (2)	119 (2)	118 (2)
N(17)–C(18)–N(19)	124 (2)	124 (2)	123 (2)
C(18)–N(19)–C(20)	120 (1)	122 (1)	119 (1)
N(19)–C(20)–C(21)	109 (1)	111 (1)	114 (1)
C(20)–C(21)–C(22)	107 (2)	110 (2)	112 (2)
C(21)–C(22)–C(23)	113 (2)	115 (2)	117 (1)
C(22)–C(23)–N(24)	113 (1)	113 (1)	114 (2)
C(23)–N(24)–C(25)	126 (1)	124 (1)	129 (2)
N(24)–C(25)–N(26)	119 (2)	123 (1)	121 (2)
N(24)–C(25)–N(27)	119 (2)	115 (2)	115 (2)
N(26)–C(25)–N(27)	122 (1)	121 (1)	124 (2)

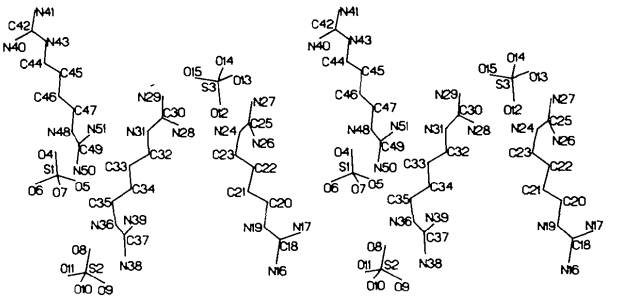

Fig. 1. Stereoview of the molecule and atom numbering.

molecule, out of which eight of molecule (I) and nine of molecules (II) and (III) are involved in N—H···O-type bonding.

The configurational similarities between sulphate and phosphate ions make it possible to extrapolate the observations made in the present study to the possible mode of binding of arcaine molecules to nucleic acids. The inter-sulphate distance of 7·26 Å along the *a* axis can be compared with that of 7·3 Å between successive phosphate groups along the helix in polynucleotides. As in the previous studies we observe that the amine molecule has the correct geometry to bridge in DNA helices through hydrogen bonds with the phosphate ions of the nucleic acids, stabilizing their structures.

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Structures of Two 2-Arylpyrazolo[4,3-*c*]quinolin-3-ones: CGS8216, $C_{16}H_{11}N_3O$, and CGS9896, $C_{16}H_{10}ClN_3O$*

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Abstract. CGS8216, 2-phenyl-2,5-dihydropyrazolo-[4,3-*c*]quinolin-3(3*H*)-one, $M_r = 261{\cdot}28$, $P2_1/c$, $a = 8{\cdot}147\ (2)$, $b = 12{\cdot}855\ (2)$, $c = 12{\cdot}521\ (3)$ Å, $\beta = 103{\cdot}62\ (2)°$, $V = 1274{\cdot}4\ (5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}36$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}08$ mm^{-1}, $F(000) = 544$, $T = 298$ K, $R = 0{\cdot}037$ for 1147 unique observed reflections. CGS9896, 2-(4-chlorophenyl)-2,5-dihydropyrazolo[4,3-*c*]quinolin-3(3*H*)-one, $M_r = 295{\cdot}73$, *Pbca*, $a = 14{\cdot}790\ (3)$, $b = 9{\cdot}515\ (1)$, $c = 18{\cdot}326\ (3)$ Å, $V = 2578{\cdot}9\ (7)$ Å^3, $Z = 8$, $D_x = 1{\cdot}52$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}29$ mm^{-1}, $F(000) = 1216$, $T = 298$ K, $R = 0{\cdot}046$ for 1117 unique observed reflections. The crystal packing of the two compounds is discussed. It is shown that intermolecular hydrogen bonding can cause small but significant changes in the geometry of the –HN–C=C–C=O fragment. The role played by these compounds as benzodiazepine-receptor ligands in the central nervous system is briefly reviewed.

Introduction. The central nervous system contains stereospecific saturable high-affinity recognition sites for benzodiazepines (BDZ's), which are thought to mediate their pharmacological effects (Squires & Braestrup, 1977; Möhler & Okada, 1977). There is evidence that BDZ's exert their action mainly *via* the GABA system (GABA = γ-aminobutyric acid) (Braestrup, Nielsen, Honoré, Jensen & Petersen, 1983), the molecular basis of this interaction being probably the GABA–BDZ-receptor-chloride channel complex. BDZ's are widely employed in therapeutics as anxiolytic and anticonvulsant agents. More recently, several new drugs have been discovered interacting with BDZ

* Stereochemistry of Benzodiazepine-Receptor Ligands. II. Part I: Bertolasi, Ferretti, Gilli & Borea (1984).

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