C3	−0.0451 (4)	0.0625 (2)	0.7684 (4)	0.041 (1)
C4	−0.0102 (4)	0.1326 (2)	0.7594 (4)	0.046 (1)
C5	0.1505 (3)	0.2192 (2)	0.8507 (5)	0.049 (1)
C6	0.2263 (4)	0.2457 (2)	0.9897 (6)	0.062 (1)
C7	0.3162 (4)	0.1954 (2)	1.0602 (5)	0.062 (2)
C8	0.2403 (3)	0.1336 (2)	1.0990 (4)	0.048 (1)
C9	0.1703 (3)	0.1059 (2)	0.9554 (4)	0.038 (1)
C10	0.0747 (3)	0.1575 (2)	0.8909 (4)	0.040 (1)
C11	0.1827 (3)	−0.0089 (2)	1.0204 (4)	0.045 (1)
C12	0.1204 (3)	−0.0736 (2)	1.0540 (4)	0.043 (1)
C13	0.1805 (4)	−0.1281 (2)	0.9907 (4)	0.054 (1)
C14	0.1260 (5)	−0.1890 (2)	1.0121 (5)	0.068 (2)
C15	0.0131 (5)	−0.1963 (2)	1.1009 (6)	0.070 (2)
C16	−0.0436 (5)	−0.1433 (2)	1.1711 (5)	0.068 (2)
C17	0.0093 (4)	−0.0822 (2)	1.1475 (5)	0.055 (1)
C21	−0.1413 (3)	0.0614 (1)	1.0438 (4)	0.038 (1)
C22	−0.1116 (4)	0.0849 (2)	1.1913 (4)	0.047 (1)
C23	−0.2121 (5)	0.1085 (2)	1.2875 (5)	0.063 (2)
C24	−0.3427 (4)	0.1079 (2)	1.2391 (5)	0.069 (2)
C25	−0.3738 (4)	0.0835 (2)	1.0951 (6)	0.066 (2)
C26	−0.2747 (3)	0.0603 (2)	0.9979 (5)	0.050 (1)

Table 2. *Selected geometric parameters* (Å, °)

O4—C4	1.210 (5)	N1—C2	1.475 (4)
O11—C11	1.235 (4)	N1—C9	1.484 (4)
O4—C4—C3	122.1 (3)	C13—C12—C17	118.2 (4)
C3—C4—C10	115.1 (3)	C2—C21—C26	118.9 (3)
O4—C4—C10	122.8 (4)	C2—C21—C22	123.1 (3)
C11—C12—C17	124.0 (4)	C22—C21—C26	118.0 (3)
C11—C12—C13	117.7 (3)		
C2—N1—C11—C12	−22.8 (5)	O4—C4—C10—C9	−148.9 (4)
C2—N1—C9—C10	11.0 (4)	C6—C5—C10—C9	−56.7 (4)
C11—N1—C9—C8	−69.1 (4)	C10—C5—C6—C7	54.0 (5)
C2—N1—C9—C8	133.7 (3)	C5—C6—C7—C8	−53.5 (5)
C9—N1—C2—C3	41.4 (4)	C6—C7—C8—C9	56.6 (4)
N1—C2—C21—C22	−6.7 (5)	C7—C8—C9—C10	−58.2 (4)
N1—C2—C3—C4	−59.2 (4)	N1—C9—C10—C4	−49.4 (4)
C3—C2—C21—C22	−131.7 (4)	C8—C9—C10—C5	58.2 (4)
C2—C3—C4—C10	21.9 (5)	N1—C11—C12—C13	139.8 (3)
C3—C4—C10—C5	158.3 (3)	O11—C11—C12—C13	−39.8 (5)
C3—C4—C10—C9	31.8 (4)		

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). All non-H atoms were refined with anisotropic displacement parameters. All H atoms were obtained from difference Fourier maps and were included in the structure-factor calculations; they were given displacement parameters equal to $1.1U_{eq}$ of their respective carrier atom, but their parameters were not refined (Sheldrick, 1976). The geometrical calculations were performed using *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, least-squares-planes data and torsion angles have been deposited with the IUCr (Reference: VJ1020). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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o-Phenylenediammonium Bis(hydrogensulfide)

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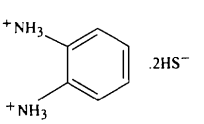
Abstract

The title compound, $C_6H_{10}N_2^{2+}.2HS^-$, forms crystals with *C*2/*c* symmetry. The anionic HS^- groups are located near the NH_3^+ substituents and are oriented practically perpendicular to the benzene ring.

Comment

The molecule in the unit cell of the title compound, (I), is situated on the twofold axis, which passes through the midpoints of the C(4*A*)—C(4*B*) and C(1*A*)—C(1*B*) bonds.



(I)

The presence of H atoms at every N atom and the C(4*A*)—N(1*A*) bond length of 1.457 (2) Å indicate protonation of both amino groups. The anionic HS^- groups are oriented practically perpendicular to the benzene ring [104 (1)°]. The short interatomic distances N(1*A*)···S(*A*) [3.11 (1) Å] and H1(N1*A*)···S(*A*) [2.13 (1) Å] confirm the existence of a strong electrostatic interaction between the dication and the anions.

There are no intermolecular distances shorter than the sum of the van der Waals radii. Fig. 1 shows a perspective view of (I), with the atom-numbering scheme.

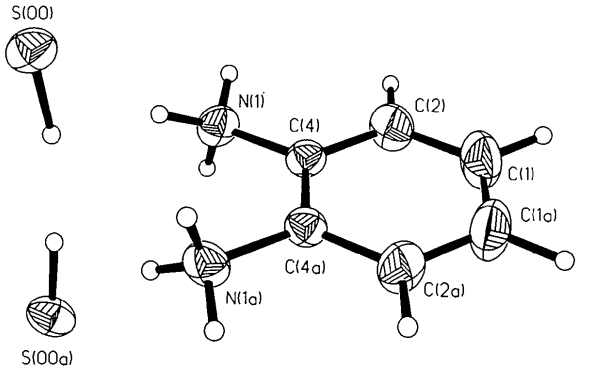


Fig. 1. A view of the title molecule with the atom-numbering scheme. Atoms are represented as 50% probability ellipsoids.

Experimental

Dark red crystals suitable for X-ray study were grown from a 2-propanol solution by slow evaporation of the solvent.

Crystal data

$C_6H_{10}N_2^{2+}.2HS^-$
$M_r = 176.31$
Monoclinic
$C2/c$
$a = 7.341\ (1)$ Å
$b = 14.518\ (3)$ Å
$c = 8.010\ (2)$ Å
$\beta = 94.01\ (3)°$
$V = 851.6\ (9)$ Å^3
$Z = 4$
$D_x = 1.375$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 9 reflections
$\theta = 12–13°$
$\mu = 0.554$ mm^{-1}
$T = 293$ K
Block
0.4 × 0.2 × 0.2 mm
Dark red

Data collection

Siemens *P3/PC* diffractometer
2θ–θ scans
Absorption correction: none
1917 measured reflections
1804 independent reflections
1119 observed reflections $[F > 6\sigma(F)]$
$R_{int} = 0.0259$
$\theta_{max} = 25°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 23$
$l = -12 \rightarrow 12$
2 standard reflections monitored every 98 reflections
intensity decay: 7%

Refinement

Refinement on F
$R = 0.037$
$wR = 0.043$
$S = 1.73$
1119 reflections
64 parameters
H atoms riding with fixed isotropic U
$w = 1/[\sigma^2(F_o) + 0.0003F_o^2]$
$(\Delta/\sigma)_{max} = 0.197$
$\Delta\rho_{max} = 0.44$ e Å^{-3}
$\Delta\rho_{min} = -0.47$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
S	0.0232 (1)	0.1767 (1)	−0.0099 (1)	0.037 (1)
N(1)	−0.1880 (2)	0.3323 (1)	−0.2069 (2)	0.035 (1)
C(1)	−0.0885 (3)	0.5828 (1)	−0.2253 (2)	0.053 (1)
C(2)	−0.1787 (2)	0.5008 (1)	−0.2017 (2)	0.042 (1)
C(4)	−0.0889 (2)	0.4181 (1)	−0.2264 (2)	0.029 (1)

Table 2. *Selected geometric parameters* (Å, °)

C(4)—C(4ⁱ)	1.384 (2)	C(4)—C(2)	1.391 (2)
C(1)—C(1ⁱ)	1.384 (4)	C(2)—C(1)	1.381 (2)
N(1)—C(4)	1.457 (2)		
N(1)—C(4)—C(2)	118.6 (1)	C(4)—C(2)—C(1)	119.2 (1)
C(2)—C(4)—C(4ⁱ)	120.3 (1)	C(2)—C(1)—C(1ⁱ)	120.5 (1)
N(1)—C(4)—C(4ⁱ)	121.1 (1)		

Symmetry codes: (i) $-x, y, -\frac{1}{2} - z$.

The structure was solved by direct methods using *SHELXTL-Plus* (Sheldrick, 1991). After non-H atoms were refined anisotropically, positions of all H atoms were located from a ΔF map and included in the refinement with fixed isotropic displacement parameters. Ten strong reflections with $(F_o - F_c)/\sigma > 4.0$ were excluded from the last refinement cycles.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: VS1009). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Muscarinic Antagonist: 8,8-Dimethyl-3′,3′-diphenylspiro(8-azoniabicyclo[3.2.1]octane-3,2′-1′,3′-dioxolane)-4′-one Iodide

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Abstract

The molecular structure of the title compound, $C_{23}H_{26}NO_3^+.I^-$, BVT44Me, has been compared to that of the related compound 8,8-dimethyl-3,3-diphenyl-

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