

Related literature. Acridines and 9-aminoacridines crystallize as salts [for example: proflavine hemisulfate hydrate (Jones & Neidle, 1975); 9-aminoacridine hydrochloride monohydrate (Talacki, Carrell & Glusker, 1974)] and invariably have the protonated ring nitrogen atom involved in intermolecular hydrogen bonding with anions and water molecules.

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Structure of a Norbornanepolysulfane: *exo*-3,4,5,6,7-Pentathia-tricyclo[7.2.1.0^{2,8}]dodecane

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Abstract. C₇H₁₀S₅, *M_r* = 254.46, monoclinic, *P*2₁/*n*, *a* = 10.167 (4), *b* = 11.092 (2), *c* = 10.278 (4) Å, β = 115.22 (2)°, *V* = 1049 (1) Å³, *Z* = 4, *D_x* = 1.61 Mg m⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 1.01 mm⁻¹, *F*(000) = 528, *T* = 293 K, final *R* = 0.0339 for 1858 observed reflections. The two alpha S atoms in the pentathiepane derivative of norbornane, obtained from the reaction of norbornene with activated sulfur [Bartlett & Ghosh (1987). *J. Org. Chem.* 52, 4937–4943], are both in *exo* positions.

Experimental. Bright yellow crystals were obtained from a solution of C₇H₁₀S₅ in *n*-pentane, saturated at 293 K and kept for 24 h at 275 K. A crystal of approximate dimensions 0.3 × 0.3 × 0.4 mm was used for the X-ray analysis. Intensities measured on a Syntex *P*2₁ diffractometer, Mo *K*α radiation (graphite monochromator). Cell dimensions determined from least-squares refinement of 25 reflections (8.7 ≤ 2θ ≤ 24.0°). Reflections measured in the ω-scan mode, 2θ_{max} = 55° (−13 ≤ *h* ≤ 11, 0 ≤ *k* ≤ 13, 0 ≤ *l* ≤ 13); two standard reflections (303; $\bar{1}$ 21) measured every 100 reflections showed only random variations ≤ 1.5%. 2610 reflections collected, 2213 symmetry independent (*R*_{int} = 0.019), 1858 observed with *I* ≥ 2σ(*I*). Lorentz and polarization corrections applied,

References

- FRENZ, B. A. (1980). *Enraf-Nonius Structure Determination Package*. Version 17. College Station, Texas, USA.
 JONES, T. A. & NEIDLE, S. (1975). *Acta Cryst.* B31, 1324–1333.
 MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCO, J.-P. & WOOLFSON, M. M. (1982). *MULTAN82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
 TALACKI, R., CARRELL, H. L. & GLUSKER, J. P. (1974). *Acta Cryst.* B30, 1044–1047.
 WALKER, N. L. & STUART, D. (1983). *Acta Cryst.* A39, 158–166.

empirical absorption correction with *DIFABS* (Walker & Stuart, 1983), minimum/maximum corrections 0.780/1.139.

Structure solved by direct methods using the program *SHELX86* (Sheldrick, 1986), refinement with *SHELX76* (Sheldrick, 1976), geometrical calculations with *PLATON* (Spek, 1982). Atomic scattering factors taken from Cromer & Mann (1968). H-atom positions calculated assuming *sp*³ hybridization and C—H bond lengths of 1.08 Å. Full-matrix least-squares refinements on *F* with anisotropic thermal parameters, unit weights, 110 parameters, *R* = 0.034; (Δ/σ)_{max} = 0.0001; Δρ_{min} −0.27, Δρ_{max} 0.34 e Å⁻³, goodness of fit = 0.71. Atomic parameters are given in Table 1 and bond lengths and angles are in Table 2.† Fig. 1 shows a view of the molecule with the atom-numbering scheme.

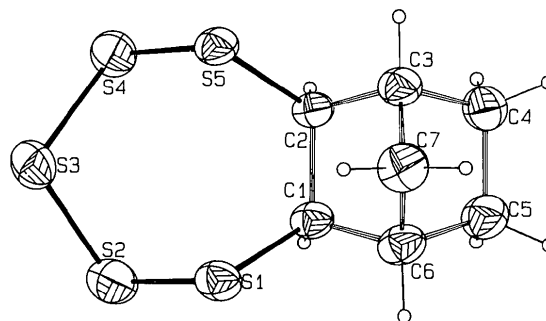
Related literature. Sulfuration of norbornene derivatives gives the trithiolane and pentathiepine derivatives. The position of both alpha S atoms is always determined as the *exo* position (Bartlett & Ghosh, 1987; Watson, Jain, Bartlett & Ghosh, 1985; Emsley & Griffiths, 1979).

† Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54468 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0255]

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Table 1. Atomic coordinates and equivalent isotropic thermal parameters (\AA^2)
$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	B_{eq}
S(1)	0.3325 (1)	0.0813 (1)	1.1465 (1)	2.91
S(2)	0.2228 (1)	0.0747 (1)	1.2719 (1)	3.61
S(3)	0.1812 (1)	0.2523 (1)	1.3020 (1)	3.43
S(4)	0.0233 (1)	0.3039 (1)	1.1042 (1)	3.31
S(5)	0.1318 (1)	0.3212 (1)	0.9784 (1)	2.72
C(1)	0.1911 (3)	0.0639 (2)	0.9633 (3)	2.37
C(2)	0.1015 (3)	0.1778 (2)	0.8829 (3)	2.20
C(3)	0.1482 (3)	0.1959 (3)	0.7598 (3)	2.76
C(4)	0.0743 (4)	0.0967 (3)	0.6477 (3)	3.25
C(5)	0.1577 (4)	-0.0187 (3)	0.7245 (3)	3.41
C(6)	0.2699 (3)	0.0285 (3)	0.8697 (3)	2.96
C(7)	0.3066 (3)	0.1526 (3)	0.8280 (4)	3.47

Fig. 1. Perspective view of the $\text{C}_7\text{H}_{10}\text{S}_5$ molecule with atom-numbering schemeTable 2. Bond distances (\AA) and angles ($^\circ$)

S(1)—S(2)	2.034 (1)	S(1)—C(1)	1.829 (3)
S(3)—S(4)	2.063 (1)	S(4)—S(5)	2.034 (1)
C(1)—C(2)	1.570 (4)	C(1)—C(6)	1.541 (4)
C(2)—C(3)	1.543 (4)	C(3)—C(4)	1.538 (4)
C(4)—C(5)	1.546 (5)	C(5)—C(6)	1.540 (4)
S(2)—S(3)	2.066 (1)	C(3)—C(7)	1.534 (5)
S(5)—C(2)	1.825 (2)	C(6)—C(7)	1.535 (5)
S(2)—S(1)—C(1)	104.3 (1)	S(1)—S(2)—S(3)	105.2 (1)
S(2)—S(3)—S(4)	103.9 (1)	S(3)—S(4)—S(5)	104.3 (1)
S(4)—S(5)—C(2)	104.3 (1)	S(1)—C(1)—C(2)	118.8 (2)
S(1)—C(1)—C(6)	106.3 (2)	C(2)—C(1)—C(6)	102.5 (2)
S(5)—C(2)—C(1)	119.4 (2)	S(5)—C(2)—C(3)	106.7 (2)
C(1)—C(2)—C(3)	102.9 (2)	C(2)—C(3)—C(4)	107.4 (2)
C(2)—C(3)—C(7)	102.4 (2)	C(4)—C(3)—C(7)	101.6 (3)
C(3)—C(4)—C(5)	103.0 (2)	C(4)—C(5)—C(6)	103.4 (3)
C(1)—C(6)—C(5)	108.3 (3)	C(1)—C(6)—C(7)	101.4 (2)
C(5)—C(6)—C(7)	101.9 (2)	C(3)—C(7)—C(6)	94.3 (3)

References

- BARTLETT, P. D. & GHOSH, T. (1987). *J. Org. Chem.* **52**, 4937–4943.
- CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* **A24**, 321–324.
- EMSLEY, J. & GRIFFITHS, D. W. (1979). *Acta Cryst.* **B35**, 2119–2121.
- SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- SHELDRICK, G. M. (1986). *SHELX86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.
- SPEK, A. L. (1982). *PLATON*. The *EUCLID* package. In *Computational Crystallography*, edited by D. SAYRE, p. 528, Oxford: Clarendon Press.
- WALKER, N. & STUART, D. (1983). *Acta Cryst.* **A39**, 158–166.
- WATSON, W. H., JAIN, P. C., BARTLETT, P. D. & GHOSH, T. (1985). *Acta Cryst.* **C42**, 332–334.

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1,3-Propanediammonium Diperchlorate

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Abstract. $\text{C}_3\text{H}_{12}\text{N}_2^{2+} \cdot 2\text{ClO}_4^-$, $M_r = 275.04$, monoclinic, $P2_1/c$, $a = 7.298$ (4), $b = 14.388$ (9), $c = 9.708$ (6) \AA , $\beta = 96.79$ (4) $^\circ$, $V = 1012.2$ \AA^3 , $Z = 4$, $D_x = 1.80$ Mg m^{-3} , $\lambda(\text{Mo K}\alpha) = 0.71073$ \AA , $\mu = 0.596$ mm^{-1} , $F(000) = 568$, $T = 293$ K, $R = 0.053$ for 1053 unique reflexions [$F \geq 3\sigma(F)$]. The crystal

structure is composed of doubly charged 1,3-propanediammonium cations and tetrahedral perchlorate anions. One N atom is surrounded by eight perchlorate O atoms and the other by seven in the weakly hydrogen-bonding range 2.95 (1)–3.23 (1) \AA . The ammonium groups have a conformation with