

Fig. 2. View of the molecule.

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Structure of a Bicyclic Sulfur–Nitrogen–Carbon Heterocyclic Molecule

BY A. W. CORDES

Department of Chemistry, University of Arkansas, Fayetteville, AR 72701, USA

AND R. T. OAKLEY AND RENÉ T. BOERÉ

Guelph–Waterloo Centre for Graduate Work in Chemistry, Guelph Campus, Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1

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Abstract. 7-Phenyl-1 λ^4 ,3 λ^4 δ^2 ,5 λ^4 -trithia-2,4,6,8,9-pentaazabicyclo[3.3.1]nonane, $C_7H_5N_5S_3$, $M_r = 255.3$, monoclinic, $P2_1/n$, $a = 5.958$ (1), $b = 22.954$ (2), $c = 7.428$ (1) Å, $\beta = 106.25$ (1)°, $V = 975.2$ (4) Å³, $Z = 4$, $D_x = 1.74$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 7.00$ cm⁻¹, $F(000) = 520$, $T = 293$ K, $R = 0.030$ for 1363 unique observed reflections. The planar SNCNS and SNSNS components of the bicyclic molecule make dihedral angles of 137.1 and 118.3° with the SNS bridging unit. The S–N bonds connecting the NSN fragment to the CN₃S₂ ring are much longer (1.728 Å) than those in the remainder of the molecule (1.546–1.630 Å).

Experimental. Compound prepared by reaction of *N,N,N'*-tris(trimethylsilyl)benzamidine with S₃N₃Cl₃. Crystals obtained from acetonitrile solutions. Yellow

needle data crystal 0.60 × 0.16 × 0.14 mm mounted on a glass fiber. Density not measured. Intensities measured with an Enraf–Nonius CAD-4 diffractometer using variable-speed ω - 2θ scans. Unit cell determined from least-squares analysis of angle data for 25 reflections with $20 < 2\theta < 28^\circ$. Analytical absorption correction based on crystal shape varied from 0.95 to 1.00. Data collected to $\sin\theta/\lambda$ of 0.59 Å⁻¹, $-7 \leq h \leq 7$, $0 \leq k \leq 27$, $0 \leq l \leq 8$. Three standard reflections (252, 1,12,0, $\bar{3}73$) varied $\pm 4.1\%$ over 16.9 h of data collection; anisotropic-drift correction applied. 1839 reflections measured, 1708 unique ($R_{\text{int}} = 0.01$), 345 reflections with $I < 3\sigma(I)$ considered unobserved. Solved by direct methods using *MULTAN11/82* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) and Fourier methods. Full-matrix least squares minimized $\sum w\Delta F^2$. H atoms refined isotropically, other

Table 1. Fractional atomic coordinates, isotropic thermal parameters, and their e.s.d.'s

$$B_{eq} = \frac{4}{3}(a^2B_{11} + b^2B_{22} + c^2B_{33} + acB_{13} \cos \beta).$$

	x	y	z	$B_{eq}/B(\text{\AA}^2)$
S(1)	0.8959 (1)	0.05740 (3)	0.67029 (8)	2.57 (1)
S(2)	0.4814 (1)	0.08893 (3)	0.72098 (9)	2.60 (1)
S(3)	0.6221 (1)	-0.02880 (3)	0.78955 (9)	2.86 (1)
N(1)	1.0132 (4)	0.0940 (1)	0.8598 (3)	2.67 (5)
N(2)	0.6415 (4)	0.1160 (1)	0.9165 (3)	2.98 (5)
N(3)	0.6345 (4)	0.0824 (1)	0.5714 (3)	2.80 (5)
N(4)	0.8576 (4)	-0.0106 (1)	0.7568 (3)	3.20 (5)
N(5)	0.4353 (4)	0.0185 (1)	0.7842 (3)	3.00 (5)
C(1)	0.8736 (4)	0.1177 (1)	0.9533 (3)	2.38 (5)
C(2)	0.9938 (4)	0.1513 (1)	1.1240 (3)	2.32 (5)
C(3)	0.8903 (5)	0.1568 (1)	1.2681 (4)	2.89 (6)
C(4)	0.9998 (6)	0.1880 (1)	1.4279 (4)	3.63 (7)
C(5)	1.2119 (6)	0.2145 (1)	1.4424 (5)	4.00 (7)
C(6)	1.3156 (5)	0.2092 (1)	1.3000 (4)	3.90 (7)
C(7)	1.2099 (5)	0.1774 (1)	1.1401 (4)	3.00 (6)
H(1)	0.743 (4)	0.141 (1)	1.256 (3)	2.5 (5)
H(2)	0.924 (4)	0.191 (1)	1.526 (3)	2.6 (6)
H(3)	1.278 (6)	0.234 (1)	1.548 (5)	5.3 (8)
H(4)	1.459 (5)	0.233 (1)	1.308 (4)	4.1 (7)
H(5)	1.275 (5)	0.177 (1)	1.036 (4)	3.5 (6)

Table 2. Selected bond distances (Å), bond angles (°), and their e.s.d.'s

S(1)-N(3)	1.629 (2)	N(3)-S(1)-N(4)	106.1 (1)
S(1)-N(4)	1.728 (2)	N(4)-S(1)-N(1)	102.5 (1)
S(1)-N(1)	1.621 (2)	N(3)-S(1)-N(1)	109.5 (1)
S(2)-N(3)	1.630 (2)	N(3)-S(2)-N(5)	105.1 (1)
S(2)-N(5)	1.728 (2)	N(3)-S(2)-N(2)	110.2 (1)
S(2)-N(2)	1.621 (2)	N(5)-S(2)-N(2)	102.3 (1)
S(3)-N(5)	1.546 (2)	N(5)-S(3)-N(4)	119.0 (1)
S(3)-N(4)	1.547 (2)	S(1)-N(3)-S(2)	111.8 (1)
N(1)-C(1)	1.339 (3)	S(2)-N(5)-S(3)	119.7 (1)
N(2)-C(1)	1.333 (3)	S(1)-N(4)-S(3)	121.5 (1)
C(1)-C(2)	1.485 (3)	S(1)-N(1)-C(1)	118.8 (2)
C-C(phenyl) range	1.372-1.394	S(2)-N(2)-C(1)	120.8 (2)
C-C(phenyl) mean	1.383	N(1)-C(1)-N(2)	129.8 (2)
C-H range	0.90-1.00	N(1)-C(1)-C(2)	115.6 (2)
C-H mean	0.95	N(2)-C(1)-C(2)	114.6 (2)
		C-C-C range	119.1-120.9
		C-C-H range	117-123

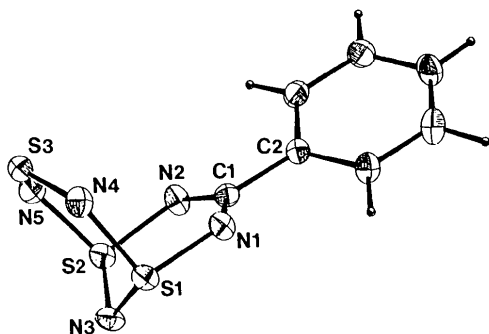


Fig. 1. ORTEP diagram (Johnson, 1976) and atom-numbering scheme. Non-H ellipsoids at 30% probability level, H atoms given arbitrary radii.

atoms anisotropically for a total of 156 variables. $R = 0.030$, $wR = 0.044$, $S = 1.36$, where non-Poisson $w^{-2} = \sigma^2(F^2) = \sigma(I) + 0.0025F^2$. Final $(\Delta/\sigma)_{\max} < 0.01$, $\Delta\rho_{\max} = 0.22$ (6) and $\Delta\rho_{\min} = -0.33$ (6) $e \text{ \AA}^{-3}$ on final difference map. Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974) and programs used those of Enraf-Nonius (1982) *SDP*.^{*} Table 1 gives the atom parameters and Table 2 bond lengths and angles. Fig. 1 shows the molecule and numbering scheme.

Related literature. Structures of three other derivatives of 5-phenyl-1,3,2,4,6-dithiaziazine are in preparation or have been published: $\text{PhCS}_2\text{N}_3\text{Cl}_2$ (Graham, Cordes, Oakley & Boeré, 1985), $\text{PhCS}_2\text{N}_3\text{C}_7\text{H}_8$ (James, Craig, Cordes, Oakley & Boeré, 1985), and $(\text{PhCS}_2\text{N}_3)_2$ (Boeré, French, Oakley, Cordes, James, Craig & Graham, 1985). The long N-S bonds to the bridging NSN can be compared to the 1.692 (8) Å values observed in the related bicyclic molecule $\text{F}_2\text{N}_5\text{S}_3$ (Weiss, Ruppert & Appel, 1974).

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