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Structure of a Bicyclic Sulfur–Nitrogen–Carbon Heterocyclic Molecule

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Abstract. 7-Phenyl-1,3,5,7,9,11-trithia-2,4,6,8,10-pentaazabicyclo[3.3.1]nonane, C₁₇H₁₅N₅S₃, Mr = 255.3, monoclinic, P2₁/n, a = 5.958 (1), b = 22.954 (2), c = 7.428 (1) Å, β = 106.25 (1)°, V = 975.2 (4) Å³, Z = 4, Dx = 1.74 g cm⁻³, λ(Mo Kα) = 0.71073 Å, μ = 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 S₃NCl₃. 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 < 2θ < 28°. Analytical absorption correction based on crystal shape varied from 0.95 to 1.00. Data collected to sin(θ)/λ of 0.59 Å⁻¹, −7 ≤ h ≤ 7, 0 ≤ k ≤ 27, 0 ≤ l ≤ 8. Three standard reflections (252, 1,12,0, 373) varied ±4.1% over 16.9 h of data collection; anisotropic-drift correction applied. 1839 reflections measured, 1708 unique (Rint = 0.01), 345 reflections with I < 3σ(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 ∑wAF². H atoms refined isotropically, other
A BICYCLIC SULFUR–NITROGEN–CARBON HETEROCYCLIC MOLECULE

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

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<th>Atom</th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>B_eq/(90^2)</th>
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<td>S(3)</td>
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<tr>
<td>N(1)</td>
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<td>0.6709(8)</td>
<td>2.57(1)</td>
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<td>N(3)</td>
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<tr>
<td>N(4)</td>
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<td>0.1880(1)</td>
<td>0.7568(3)</td>
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<tr>
<td>N(5)</td>
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<td>0.0185(1)</td>
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Table 2. Selected bond distances (Å), bond angles (°), and their e.s.d.'s.

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<th>Bond</th>
<th>Bond Angle</th>
<th>e.s.d.</th>
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<td>S(1)--N(4)</td>
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<tr>
<td>S(1)--N(1)</td>
<td>1.339 (3)</td>
<td>0.020</td>
</tr>
<tr>
<td>S(2)--N(5)</td>
<td>1.630 (2)</td>
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<tr>
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<td>1.546 (2)</td>
<td>0.015</td>
</tr>
<tr>
<td>S(3)--N(5)</td>
<td>1.546 (2)</td>
<td>0.015</td>
</tr>
<tr>
<td>N(1)--C(1)</td>
<td>1.339 (3)</td>
<td>0.020</td>
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<tr>
<td>N(2)--C(1)</td>
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<tr>
<td>C(1)--C(3) mean</td>
<td>1.372–1.394</td>
<td>0.020</td>
</tr>
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Fig. 1. ORTEPII diagram (Johnson, 1976) and atom-numbering scheme. Non-H ellipsoids at 30% probability level, H atoms given arbitrary radii.