

SHORT-FORMAT PAPERS

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Acta Cryst. (1985). C41, 1686–1687

Structure of an Eight-Membered CN₄S₃ Ring with the Shortest Known Transannular S...S Contact

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(Received 2 July 1985; accepted 22 July 1985)

Abstract. 7-*tert*-Butyl-3-chloro-1,3,5,2,4,6,8-trithiatetrazocine, C₅H₉ClN₄S₃, $M_r = 256.8$, tetragonal, $P4_2/n$, $a = 17.856$ (2), $c = 6.646$ (1) Å, $V = 2118.9$ (8) Å³, $Z = 8$, $D_x = 1.61$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 8.9$ cm⁻¹, $F(000) = 1056$, $T = 294$ K, final $R = 0.049$ for 1201 reflections. The molecule has a folded butterfly structure, with almost planar SNCNS and SNSNS fragments, and the Cl atom is *endo*. The transannular S–S contact is 2.378 (2) Å, the shortest such distance in any thiazyl compound.

Experimental. Yellow needles crystallized from acetonitrile obtained from the reaction of trimethylacetamide and S₃N₃Cl₃. Data crystal 0.30 × 0.30 × 0.52 mm. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated radiation using θ – 2θ scans. Unit cell determined from 25 reflections, $20^\circ < 2\theta < 26^\circ$. Data collected to $\sin\theta/\lambda = 0.56$ Å⁻¹, h 0→21, k 0→21, l 0→7, 2010 reflections measured, 1840 unique ($R_{\text{int}} = 0.02$), 1201 reflections with $I > 3\sigma(I)$ used in refinement. Standard reflections 910, 581, 224, total decay 9.7%. Absorption correction ranged from 0.76 to 0.79. Structure solved by direct methods and refined by full-matrix least-squares techniques which minimized $\sum w\Delta F^2$. At least one H atom of each methyl group located on a difference map. H atoms constrained to idealized positions (C–H = 0.95 Å) with isotropic thermal parameters equal to 1.2 times that of the C to which they are attached. 118 parameters refined with reflections/parameters ratio 10.2/1. $R = 0.049$, $wR = 0.061$, $S = 1.64$. Final $(\Delta/\sigma)_{\text{max}} = 0.001$, $\Delta\rho = -0.041$ e Å⁻³. Weighting scheme based on counting

statistics ($p = 0.05$), and showed no dependence of $\Delta F/\sigma$ on either the magnitude of F or the value of θ . Atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). All calculations performed with the Enraf–Nonius SDP package (Enraf–Nonius, 1982). Atomic coordinates and equivalent isotropic temperature factors are given in Table 1.* Selected bond distances and angles are presented in Table 2. An ORTEP illustration of the molecule is given in Fig. 1.

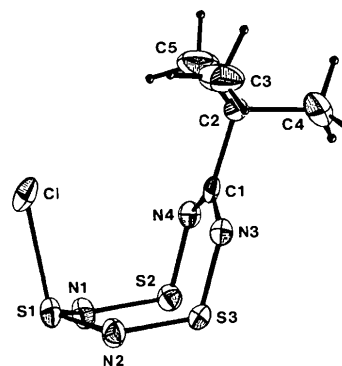
* Lists of structure factors and anisotropic temperature factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42395 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic positional ($\times 10^4$) and equivalent isotropic thermal parameters

	x	y	z	$B_{\text{eq}}(\text{Å}^2)$
Cl	386.0 (8)	2230.6 (9)	3751 (2)	4.49 (3)
S(1)	1172.9 (8)	1520.3 (8)	2011 (2)	3.53 (3)
S(2)	350.9 (7)	1362.2 (7)	-1497 (2)	3.20 (3)
S(3)	1269.8 (7)	2326.0 (7)	-1548 (2)	3.04 (3)
N(1)	641 (2)	1026 (2)	655 (7)	3.6 (1)
N(2)	1619 (2)	2081 (3)	626 (6)	3.38 (9)
N(3)	624 (2)	2940 (2)	-1337 (6)	2.72 (8)
N(4)	-283 (2)	1992 (2)	-1235 (6)	2.82 (8)
C(1)	-83 (3)	2705 (3)	-1142 (7)	2.6 (1)
C(2)	-684 (3)	3298 (3)	-1029 (8)	3.2 (1)
C(3)	-405 (4)	3975 (4)	12 (1)	6.8 (2)
C(4)	-848 (4)	3526 (4)	-320 (1)	6.1 (2)
C(5)	-1385 (4)	2990 (4)	-8 (1)	7.6 (2)

Table 2. *Interatomic distances (Å) and angles (°)*

Cl—S(1)	2.218 (2)	N(3)—C(1)	1.337 (4)
S(1)—N(1)	1.579 (4)	N(4)—C(1)	1.323 (4)
S(1)—N(2)	1.575 (4)	C(1)—C(2)	1.506 (5)
S(2)—N(1)	1.636 (4)	C(2)—C(3)	1.508 (7)
S(2)—N(4)	1.605 (3)	C(2)—C(4)	1.527 (6)
S(3)—N(2)	1.633 (3)	C(2)—C(5)	1.501 (7)
S(3)—N(3)	1.596 (3)	S(2)—S(3)	2.378 (2)
Cl—S(1)—N(1)	103.7 (1)	N(3)—C(1)—N(4)	123.6 (3)
Cl—S(1)—N(2)	105.2 (2)	N(3)—C(1)—C(2)	117.1 (3)
N(1)—S(1)—N(2)	109.0 (2)	N(4)—C(1)—C(2)	119.1 (3)
N(1)—S(2)—N(4)	112.7 (2)	C(1)—C(2)—C(3)	110.9 (4)
N(2)—S(3)—N(3)	112.5 (2)	C(1)—C(2)—C(4)	106.1 (3)
S(1)—N(1)—S(2)	118.9 (2)	C(1)—C(2)—C(5)	110.9 (4)
S(1)—N(2)—S(3)	119.6 (2)	C(3)—C(2)—C(4)	109.2 (4)
S(3)—N(3)—C(1)	118.3 (3)	C(3)—C(2)—C(5)	110.3 (5)
S(2)—N(4)—C(1)	119.3 (3)	C(4)—C(2)—C(5)	109.3 (5)

Fig. 1. ORTEP illustration (Johnson, 1976) of $t\text{-BuC(NSN)}_2\text{SCl}$.

Related literature. The molecular shape of $t\text{-BuC(NSN)}_2\text{SCl}$ may be compared to that of $(\text{CH}_3)_2\text{NC(NSN)}_2\text{CN}(\text{CH}_3)_2$ (Ernest, Holick, Rihs, Schomburg, Shoham, Wenkert & Woodward, 1981), with $d(\text{S—S}) = 2.482 \text{ \AA}$, and that of $\text{S}_4\text{N}_4\text{Cl}_2$, $d(\text{S—S}) = 2.484 (1) \text{ \AA}$ (Zak, 1981). Similar folded structures are found for the following: $R_2\text{P(NSN)}_2\text{PR}_2$, $R = \text{Me}$, $d(\text{S—S}) = 2.551 (2) \text{ \AA}$ (Burford, Chivers, Coddling & Oakley, 1982); $R = \text{Ph}$, $d(\text{S—S}) = 2.528 (1) \text{ \AA}$ (Burford, Chivers & Richardson, 1983); and $\text{Ph}_3\text{PN(NSN)}_2\text{NPPH}_3$, $d(\text{S—S}) = 2.452 (2) \text{ \AA}$ (Bojes, Chivers, Cordes, Maclean & Oakley, 1981). For comparison, $d(\text{S—S})$ in S_4N_4 is 2.58 \AA (Sharma & Donohue, 1963; De Lucia & Coppens, 1978.)

We thank the National Science Foundation, the State of Arkansas and the Natural Sciences and Engineering Research Council of Canada for financial support. RTB acknowledges receipt of an NSERC postdoctoral fellowship.

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Acta Cryst. (1985). **C41**, 1687–1689

Structure of Ethyl $\{(1S,5R,8R)\text{-8-Hydroxy-3-oxo-2-oxa-6-azabicyclo[3.3.0]oct-6-yl}\}$ acetate Hydrobromide

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(Received 4 June 1985; accepted 22 July 1985)

Abstract. $\text{C}_{10}\text{H}_{16}\text{NO}_5^+\text{Br}^-$, $M_r = 310.15$, orthorhombic, $P2_12_12_1$, $a = 5.5495 (6)$, $b = 12.3923 (15)$, $c = 18.8252 (21) \text{ \AA}$, $V = 1294.6 (3) \text{ \AA}^3$, $Z = 4$, $D_x = 1.591 \text{ Mg m}^{-3}$, $\text{Mo K}\alpha$, $\lambda = 0.7107 \text{ \AA}$, $\mu =$

3.372 mm^{-1} , $F(000) = 632$, $T = 294 \text{ K}$, final $R = 0.047$ for 1100 reflections. The hydrobromide of an intermediate in the enantioselective synthesis of the pyrrolizidine-triol crotanecine was examined to confirm its stereochemistry. There are no unusual bond distances or angles.

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