

The cyclooctene ring has a twist-boat conformation, with torsion angles ω_1 C3—C2—C1—C8, C4—C5—C6—C7, ω_2 C2—C1—C8—C7, C1—C2—C3—C4, C3—C4—C5—C6, C5—C6—C7—C8 and ω_3 C2—C3—C4—C5, C1—C8—C7—C6 of $\omega_1 = -2.8$ (2), $\omega_2 = 58.4$ (2), -85.5 (2) and $\omega_3 = 33.8$ (2), 18.5 (2) $^\circ$ that are distorted from the ideal boat D_{2d} conformation (of cyclooctane) having torsion angles of $\omega_1 = \pm 0$, $\omega_2 = \pm 76.4$ and $\omega_3 = \pm 0^\circ$ (Hendrickson, 1964), and $\omega_1 = \pm 0$, $\omega_2 = \pm 73.4$, and $\omega_3 = \pm 0^\circ$ (Ivanov & Osawa, 1984).

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5-Methyl-4-(1-piperidyl)-5-trifluoromethyl-1,2-oxathiole

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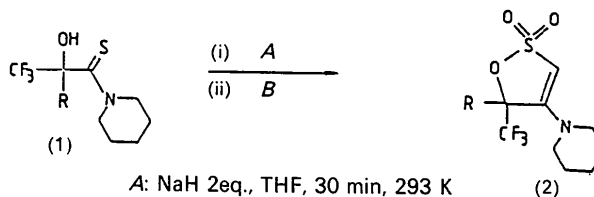
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Abstract. $C_{10}H_{14}F_3NO_3S$, $M_r = 285.28$, orthorhombic, $Pca2_1$, $a = 9.324$ (2), $b = 12.038$ (3), $c = 11.252$ (3) Å, $V = 1263.0$ (5) Å³, $Z = 4$, $D_x = 1.50$ g cm⁻³, $Mo K\alpha$, $\lambda = 0.71069$ Å, $\mu = 2.97$ cm⁻¹, $F(000) = 592$, $T = 291$ K, $R = 0.032$ for 1124 observed reflections. The five-membered unsaturated sultone ring is slightly nonplanar adopting a flat C2 half-chair conformation with endocyclic torsion angles of 5.2 (8), -1.9 (8), -1.8 (8), 5.1 (8) and -6.6 (8) $^\circ$. The endocyclic S—O and S—C bond lengths are 1.599 (2) and 1.689 (4) Å, respectively, and the C—S—O angle is 95.4 (2) $^\circ$.

Experimental. The reaction of methanesulfonyl chloride with the sodium alcoholate of the 2-hydroxythioamide (1), in the presence of an excess of sodium hydride, leads unexpectedly to the product

(2) (Maliverney, Merenyi & Viehe, 1990). This compound was identified as being a sultone, *i.e.* a cyclic sulfonate and the X-ray analysis was undertaken in order to confirm this structure and to determine the geometry of this new unsaturated heterocycle.



A: NaH 2eq., THF, 30 min, 293 K
 B: Cl—SO₂—CH₃ 1.2 eq., 24 h, 293 K

Parallelepiped crystal of approximate dimensions $0.35 \times 0.22 \times 0.14$ mm. Lattice parameters refined

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors (\AA^2)
$$B_{eq} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	B_{eq}
O1	1570 (2)	8893 (2)	1473 (3)	3.96 (4)
S2	209 (1)	9609 (1)	1873 (0)	3.57 (1)
C3	-967 (3)	8950 (3)	975 (4)	3.55 (6)
C4	-352 (3)	8167 (2)	299 (3)	3.04 (5)
C5	1288 (3)	8122 (3)	515 (3)	3.09 (5)
O6	-22 (3)	9424 (3)	3106 (3)	5.69 (6)
O7	475 (3)	10725 (2)	1519 (3)	5.52 (6)
C8	1767 (4)	7014 (3)	1057 (4)	4.35 (7)
F9	1012 (3)	6758 (3)	2009 (4)	7.23 (6)
F10	1634 (4)	6180 (2)	303 (4)	7.51 (6)
F11	3108 (3)	7043 (3)	1385 (3)	6.35 (5)
C12	2225 (4)	8478 (4)	-527 (4)	4.68 (8)
N13	-1052 (3)	7455 (3)	-426 (3)	3.74 (5)
C14	-2618 (5)	7588 (4)	-489 (6)	5.14 (8)
C15	-3345 (5)	6525 (5)	-873 (6)	5.76 (10)
C16	-2736 (6)	6066 (5)	-1995 (6)	6.22 (10)
C17	-1131 (5)	5936 (4)	-1869 (6)	5.44 (9)
C18	-429 (4)	7012 (3)	-1530 (4)	4.03 (6)

using 15 reflections in the range $3 \leq 2\theta \leq 25^\circ$. Syntax $P2_1$ four-circle diffractometer, graphite-monochromatized Mo $K\alpha$ radiation. 1306 hkl independent reflections with $\sin\theta/\lambda \leq 0.62 \text{ \AA}^{-1}$; $0 \leq h \leq 11$, $0 \leq k \leq 14$, $0 \leq l \leq 13$, 1124 with $I \geq 2.5\sigma(I)$. Standard reflection 410 checked every 50 reflections: no significant deviation. Structure solved by *SHELXS86* (Sheldrick, 1985). All H atoms from difference Fourier synthesis. Anisotropic least-squares refinement (*SHELXL76*; Sheldrick, 1976) using F ; H atoms isotropic with common refined temperature factor. 205 refined parameters. $w = 1/(\sigma^2 + 0.00233F^2)$, $R = 0.032$, $wR = 0.037$ for 1124 observed reflections. Final max. shift/e.s.d. 0.15. $S = 0.84$. Max. and min. heights in final difference Fourier synthesis = 0.15 and -0.18 e \AA^{-3} . Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

The atomic parameters are given in Table 1.* Fig. 1 is a stereoscopic view of the molecule, showing the atom-numbering scheme (*PLUTO*; Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2.

Related literature. Structural data concerning cyclic sultones are relatively sparse. This compound is the first five-membered cyclic sultone with unsaturation in the 3–4 position for which a crystal structure is reported (Cambridge Structural Database; Allen *et al.*, 1979). The most similar molecule, 3,3'-tetra-

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54009 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond distances (\AA) and angles ($^\circ$)

O1—S2	1.599 (2)	C3—S2	1.689 (4)
O6—S2	1.421 (4)	O7—S2	1.423 (3)
C5—O1	1.447 (4)	C4—C5	1.549 (4)
C8—C5	1.533 (5)	C12—C5	1.524 (5)
C3—C4	1.339 (5)	N13—C4	1.352 (4)
F9—C8	1.318 (6)	F10—C8	1.320 (5)
F11—C8	1.305 (4)	C14—N13	1.471 (6)
C18—N13	1.471 (5)	C15—C14	1.510 (7)
C16—C15	1.490 (8)	C17—C16	1.511 (7)
C18—C17	1.501 (5)		
C3—S2—O1	95.4 (2)	O6—S2—O1	108.0 (2)
O6—S2—C3	114.3 (2)	O7—S2—O1	107.0 (2)
O7—S2—C3	112.9 (2)	O7—S2—O6	116.6 (2)
C5—O1—S2	114.3 (2)	C4—C5—O1	105.9 (2)
C8—C5—O1	102.1 (3)	C8—C5—C4	112.4 (3)
C12—C5—O1	106.8 (3)	C12—C5—C4	115.8 (3)
C12—C5—C8	112.6 (3)	C3—C4—C5	111.0 (3)
N13—C4—C5	123.3 (3)	N13—C4—C3	125.6 (3)
C4—C3—S2	113.1 (2)	F9—C8—C5	111.8 (3)
F10—C8—C5	112.3 (3)	F10—C8—F9	107.1 (4)
F11—C8—C5	111.6 (4)	F11—C8—F9	106.7 (4)
F11—C8—F10	107.0 (3)	C14—N13—C4	116.1 (3)
C18—N13—C4	123.3 (3)	C18—N13—C14	113.0 (3)
C15—C14—N13	111.5 (4)	C16—C15—C14	112.7 (5)
C17—C16—C15	109.6 (4)	C18—C17—C16	111.5 (4)
C17—C18—N13	110.8 (4)		

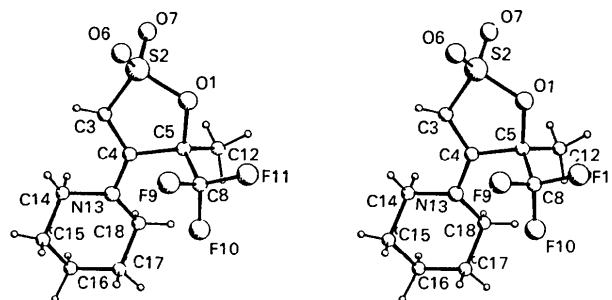


Fig. 1. Stereoscopic view of the molecule.

bromophenolsulfonphthalein, has a benzo system fused in this position (Veerapandian, Salunke & Vijayan, 1984). Its geometry is similar to the title compound with endocyclic S—O and S—C bond lengths of 1.587 and 1.736 \AA , respectively, and a C—S—O angle of 94.1° . Nevertheless the five-membered ring is less planar, the torsion around the S—O bond being 19° compared to 5° in the title compound.

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1,6,7,7-Tetrachloro-5-methyl-2-phenyl-3-azabicyclo[4.1.0]heptan-4-one

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Abstract. C₁₃H₁₁Cl₄NO, *M_r* = 339.05, triclinic, *P* $\bar{1}$, *a* = 8.180 (1), *b* = 8.318 (1), *c* = 11.542 (2) Å, α = 87.85 (1), β = 96.32 (2), γ = 110.27 (2)°, *V* = 732.2 (2) Å³, *Z* = 2, *D_x* = 1.54 g cm⁻³, *Mo K α* , λ = 0.71069 Å, μ = 7.99 cm⁻¹, *F*(000) = 344, *T* = 291 K, *R* = 0.031 for 1862 observed reflections. The configuration of the bicyclic system is *exo*. The six-membered piperidinone adopts a slightly distorted boat conformation with endocyclic torsion angles of -50 (1), 57 (1), -7 (1), -47 (1), 48 (1) and 0 (1)°. The three cyclopropane bonds have very similar lengths: 1.520 (3), 1.511 (3) and 1.521 (3) Å. Dimers are formed through hydrogen bonds between the piperidyl hydrogen and the carbonyl oxygen: N...O = 2.908 (3), H...O = 2.08 (2) Å and N—H...O = 168.5 (2)°.

Experimental. The title compound results from a [4 + 2] cycloaddition of 1-phenyl-3-trimethylsilyloxy-2-aza-1,3-pentadiene to tetrachlorocyclopropene. Since stereochemical assignments based on ¹H NMR coupling constants were uncertain, an X-ray analysis was undertaken in order to assess the configuration of the bicyclic system.

Crystals were obtained by evaporation from acetonitrile. *D_m* not measured. Parallelepiped crystal of dimensions 0.22 × 0.20 × 0.40 mm. Lattice parameters refined using 17 reflections in the range 5 ≤ 2θ ≤ 25°. Huber four-circle diffractometer, monochromatized *Mo K α* radiation. ω scans. 2168 *h* ± *k* ± *l* independent reflections with sinθ/λ ≤ 0.56 Å⁻¹; 0 ≤ *h* ≤ 9, -9 ≤ *k* ≤ 9, -12 ≤ *l* ≤ 12, 1862 with *I* ≥ 2.5σ(*I*). Standard reflection 114 checked every 50

Table 1. Atomic coordinates (× 10⁴) and equivalent isotropic temperature factors (Å²)

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B_{eq}</i>
C1	2748 (3)	7472 (3)	2365 (2)	2.95 (4)
C2	3108 (3)	8555 (3)	1255 (2)	2.81 (4)
N3	1549 (2)	9030 (3)	928 (2)	3.09 (3)
C4	1015 (3)	9868 (3)	1687 (2)	3.04 (4)
C5	1997 (3)	10067 (3)	2905 (2)	2.96 (4)
C6	2124 (3)	8325 (3)	3282 (2)	3.05 (4)
C7	3878 (3)	8052 (3)	3503 (2)	3.55 (4)
C18	1743 (1)	5275 (1)	2117 (1)	4.23 (1)
C19	5839 (1)	9814 (1)	3523 (1)	4.73 (1)
C110	4120 (1)	6519 (1)	4513 (1)	5.60 (2)
C111	438 (1)	7066 (1)	4083 (1)	4.67 (1)
C12	1234 (4)	10905 (4)	3752 (2)	3.87 (5)
O13	-149 (2)	10478 (3)	1432 (1)	4.33 (4)
C14	3625 (3)	7731 (3)	277 (2)	3.06 (4)
C15	5334 (3)	7728 (3)	354 (2)	3.63 (5)
C16	5871 (4)	6996 (3)	-517 (3)	4.36 (5)
C17	4735 (4)	6286 (4)	-1479 (3)	4.71 (6)
C18	3057 (4)	6283 (4)	-1566 (3)	5.46 (7)
C19	2488 (4)	6994 (4)	-689 (2)	4.51 (5)

reflections: no significant deviation. Structure solved by *SHELXS86* (Sheldrick, 1985). All H atoms from difference Fourier synthesis; anisotropic least-squares refinement (*SHELXL76*; Sheldrick, 1976) using *F*; H atoms isotropic with common refined temperature factor. $w = 1/(\sigma^2 + 0.00096F^2)$. *R* = 0.031, *wR* = 0.038 for 1862 observed reflections. Final max. shift/e.s.d. = 0.32 (*U*₂₂ of C12). *S* = 1.30. Max. and min. heights in final difference Fourier synthesis = 0.23 and -0.18 e Å⁻³. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).