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# 6-Chloro-8-thia-1,4-epoxybicyclo[4.3.0]non-2-ene

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In the structure of the title compound, $C_8H_9ClOS$, the six-membered ring has a boat conformation and the S-containing five-membered ring has an envelope conformation. The molecules are linked only by weak van der Waals interactions.

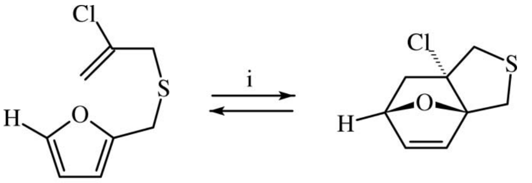
**Key indicators**

Single-crystal X-ray study
*T* = 293 K
Mean $\sigma$(C–C) = 0.004 Å
*R* factor = 0.047
*wR* factor = 0.127
Data-to-parameter ratio = 11.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

## Comment

Five-membered heteroaromatic compounds such as furans, thiophenes and pyroles possess dienic reactivity and have been well documented in the literature (Lipshutz, 1986; Kappe *et al.*, 1997). Furans, in particular, take part in inter- and intramolecular Diels–Alder reactions with a variety of dienophiles. The intramolecular Diels–Alder (IMDA) reactions of furan is particularly attractive as two, three or more rings can be constructed in a single step with high regio- and stereocontrol, providing convenient entry into natural products and the synthesis of polycyclic structures (Keay & Hunt, 1999; Demircan & Parsons, 2002; Williams, 2002). We have recently described and reported a bromo Diels–Alder cycloadduct (Büyükgüngör *et al.*, 2005). Now we outline the synthesis and crystal structure of the title compound, (2), isolated from the thermal cycloaddition of (1) in toluene in reasonably good yield. The IMDA reaction of furans undergoes a retro-cycloaddition; when the reaction is cooled to room temperature, part of the cycloadduct, (2), transforms back to (1).



(1) (2)

In general, reactions were conducted in hot toluene and the cycloaddition process is promoted by the Thorpe–Ingold (Scissor) effect. The relative stereochemistry of the cycloadduct, (2), is expected to be that of the previous examples, *i.e.* arising from an '*exo*' (the substituent on the dienophile is directed away from the diene) orientation 0.94(4)-1.06(4)of the dienophile side chain (Sammes & Weller, 1995; Parker *et al.*, 1978).

The molecular structure of (2) is shown in Fig. 1. The-six membered ring is in a boat conformation. The S-containing five-membered ring adopts an envelope conformation, with C1 deviating from the plane of the remaining four atoms by 0.528 (2) Å. The molecules are linked only by weak van der Waals interactions. The crystal structure of a closely...-compound, *viz.* 6-bromo-8-thia-1,4-epoxybicyclo[4.3.0]non-2-ene has been reported (Büyükgüngör *et al.*, 2005).

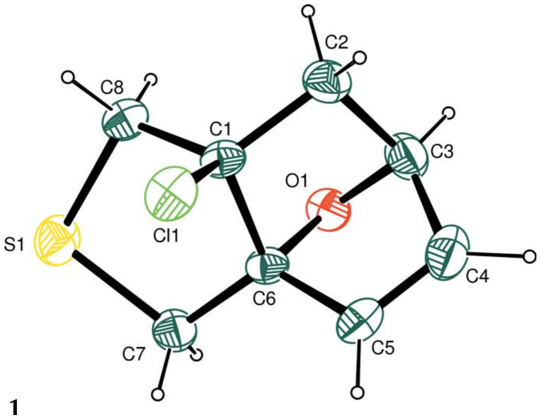


**Figure 1**
A view of (2), showing the atom-numbering scheme and 30% probability displacement ellipsoids.

## Experimental

Simple furanyl sulfides have been prepared *via* alkylation of furfuryl mercaptan; a sodium hydride suspension (0.03 g, 1.2 mmol) dehydrogenated the mercaptanol (0.09 g, 0.8 mmol); dropwise addition of 2,3-dichloropropene (0.09 g, 0.8 mmol) in tetrahydrofuran (10 ml) at 273 K afforded the precursor (1) quantitatively (yield 0.12 g, 78%). Compound (1) (0.12 g, 0.6 mmol) was then refluxed in 10 ml toluene (383 K) for 4 d. The reaction was monitored by thin layer chromatography and halted when no further change of (1) to cycloadduct (2) was noted. The ratio of furan starting material and cycloadduct was calculated after purification by flash column chromatography. The yield of cycloaddition can increase to 70% when the recovered starting material is repeatedly used for the same reaction.

### *Crystal data*

$C_8H_9ClOS$
$M_r = 188.66$
Triclinic, $P\bar{1}$
$a = 6.651$ (3) Å
$b = 7.971$ (3) Å
$c = 8.048$ (3) Å
$\alpha = 80.33$ (3)°
$\beta = 89.07$ (3)°
$\gamma = 81.43$ (3)°
$V = 415.9$ (3) Å$^3$
$Z = 2$
$D_x = 1.507$ Mg m$^{-3}$
Mo $K\alpha$ radiation
$\mu = 0.64$ mm$^{-1}$
$T = 293$ (2) K
Prism, colorless
0.43 × 0.34 × 0.17 mm

### *Data collection*

Stoe IPDS-2 diffractometer
$\omega$ scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
$T_{\min} = 0.769$, $T_{\max} = 0.896$
3731 measured reflections
1624 independent reflections
1330 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.106$
$\theta_{\max} = 26.0°$

### *Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.127$
$S = 1.06$
1624 reflections
136 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0578P)^2 + 0.0767P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.30$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.50$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| C1—Cl1 | 1.805 (2) | C8—S1 | 1.812 (3) |
| C7—S1 | 1.823 (3) | | |
| C1—C2—C3—C4 | −73.0 (3) | C3—C4—C5—C6 | 1.0 (3) |
| C2—C3—C4—C5 | 72.9 (3) | C4—C5—C6—C1 | −71.2 (3) |

All H-atom parameters were freely refined. $U_{\rm iso}$ values are in the range 0.47–0.80 Å$^2$. The C—H distances are in the range 0.94 (4)–1.06 (4) Å.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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