

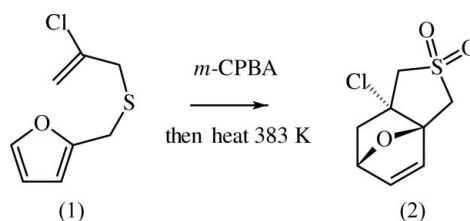
Başak Koşar,^{a*} Ersen Göktürk,^b
Aydın Demircan^b and Orhan
Büyükgüngör^a^aDepartment of Physics, Ondokuz Mayıs
University, TR-55139 Samsun, Turkey, and
^bDepartment of Chemistry, Nigde University,
TR-51100 Nigde, Turkey

Correspondence e-mail: bkosar@omu.edu.tr

Key indicators

Single-crystal X-ray study
 $T = 293$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.037
 wR factor = 0.114
Data-to-parameter ratio = 15.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.5-Chloro-10-oxa-3-thiatricyclo[5.2.1.0^{1,5}]dec-8-ene
3,3-dioxideThe title compound, $\text{C}_8\text{H}_9\text{ClO}_3\text{S}$, has been synthesized from 2-(2-chloroallylsulfanyl)methylfuran by intramolecular thermal cycloaddition. The boat form of the six-membered ring is almost symmetric with respect to the epoxy bridge, and the five-membered ring containing the S atom has an envelope conformation.Received 2 August 2006
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Comment

Alkenyl sulfones have long been known for their synthetic utility in organic chemistry, easily participating in 1,4-addition reactions and cycloaddition reactions. Their chemistry has now grown into an important branch both in heteroatom and heterocyclic chemistry from synthetic, mechanistic, structural and theoretical points of views (Thiemann *et al.*, 2000; Nenajdenko *et al.*, 2001). The sulfonyl functional group has also recently been shown to potentially inhibit a variety of enzymatic processes providing unique properties for drug design and medicinal chemistry (Supuran *et al.*, 2003). We have been studying hetero tri- and tetracycles of furan-cored compounds using intramolecular Diels–Alder cycloaddition (Demircan & Parsons, 2002). The present article focuses mainly on the X-ray crystal structure of the title compound, (2), and describes our further findings on this aspect: synthesis of tricyclic sulfone (2) from acyclic species (1) by oxidation, and subsequent thermal cycloaddition.

The molecular structure of (2) is demonstrated in Fig. 1, and the selected bond distances and angles are shown in Table 1. The six-membered ring (C1–C6) is in a boat form, which is almost symmetric with respect to the C3–O1–C6 bridge as seen from the torsion angles (Table 1). The five-membered ring adopts a distorted envelope conformation; atom C1 deviates from the C6/C7/S1/C8 plane by 0.235 (2) Å.

Conclusively, sulfones might be good candidates because of their unreactive nature towards enzymes like serine proteases and metalloproteases (Gotz *et al.*, 2004). The compounds can also be potent inhibitors of HIV-1 integrase as vinyl sulfones used in the literature (Meadows *et al.*, 2005). Tricyclic sulfones might be a new approach for the synthesis of sterically crowded fused ring systems. The ability to obtain a quaternary

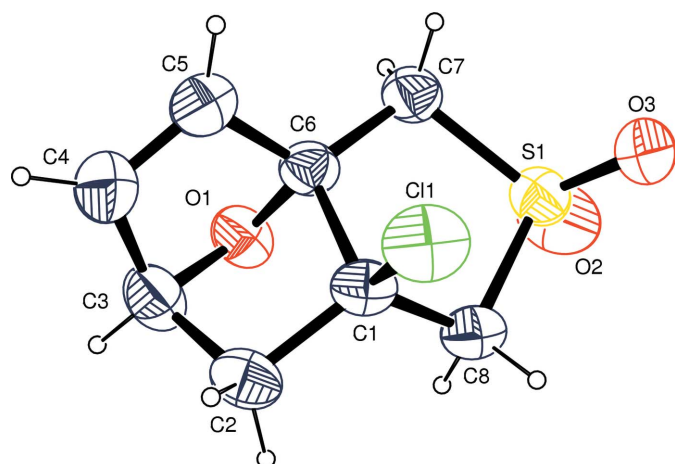


Figure 1
Molecular structure of (2), with the atom-numbering scheme and 50% probability displacement ellipsoids.

carbon originating in the dienophile and its oxidation can significantly extend the scope of the furan-based reaction with multifunctional groups.

Experimental

To a solution of *m*-chloroperbenzoic acid (*m*-CPBA) (300 mg, 1.2 mmol), which was previously purified and recrystallized from dry diethyl ether, in dichloromethane (10 ml), cooled to 273 K, was added dropwise a solution of 2-(2-chloro-allylsulfanyl)methyl-furan, (1) (0.6 mmol), in dichloromethane (10 ml) over a period of 3 min. The reaction mixture was stirred at room temperature for 4 h and diluted with cold 4% sodium bicarbonate solution (4 ml). The organic layer was separated, washed with water (20 ml) and concentrated *in vacuo*. The crude residue was heated in toluene for a further 24 h at 383 K. The solvent was then removed under reduced pressure; the resulting solid was subjected to flash column chromatography to afford compound (2) (yield 63%).

Crystal data

$C_8H_9ClO_3S$
 $M_r = 220.66$
 Triclinic, $P\bar{1}$
 $a = 6.9369$ (9) Å
 $b = 8.1298$ (11) Å
 $c = 8.3373$ (11) Å
 $\alpha = 77.625$ (10)°
 $\beta = 83.344$ (10)°
 $\gamma = 86.951$ (10)°

$V = 455.97$ (10) Å³
 $Z = 2$
 $D_x = 1.607$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.62$ mm⁻¹
 $T = 293$ (2) K
 Plate, colorless
 $0.50 \times 0.41 \times 0.06$ mm

Data collection

Stoe IPDS-2 diffractometer
 ω scans
 Absorption correction: integration
 (*X-RED*; Stoe & Cie, 2002)
 $T_{\min} = 0.746$, $T_{\max} = 0.964$

7517 measured reflections
 1786 independent reflections
 1640 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.065$
 $\theta_{\text{max}} = 26.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.037$
 $wR(F^2) = 0.114$
 $S = 1.20$
 1786 reflections
 118 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.064P)^2 + 0.0763P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\text{max}} < 0.001$$

$$\Delta\rho_{\text{max}} = 0.35 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\text{min}} = -0.47 \text{ e } \text{Å}^{-3}$$

Table 1

Selected geometric parameters (Å, °).

C1—C11	1.794 (2)	C8—S1	1.791 (2)
C3—O1	1.457 (3)	O2—S1	1.4327 (19)
C6—O1	1.439 (2)	O3—S1	1.4333 (17)
C7—S1	1.788 (2)		
C6—O1—C3	95.38 (16)		
C6—C1—C2—C3	4.6 (2)	C2—C3—C4—C5	72.9 (3)
C1—C2—C3—O1	33.3 (2)	C3—C4—C5—C6	1.7 (3)
C1—C2—C3—C4	−73.0 (2)	C4—C5—C6—O1	30.8 (2)

All H atoms were treated using a riding model, with C—H = 0.93–0.98 Å and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED* (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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