Convenient One-pot Synthesis of Cyclopropyl Sulfones *via* Successive Alkylation and Cyclization of Sulfone-stabilized Carbanions

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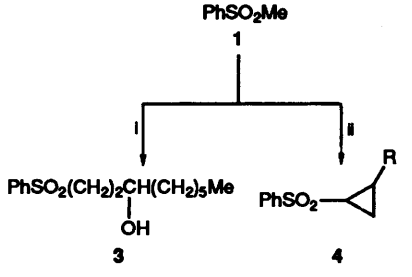
A general one-pot approach to cyclopropyl sulfones has been demonstrated, which involves the alkylation of lithiomethyl phenyl sulfone with an epoxide followed by cyclization of the resulting carbanion. Alkylation of the dianion derived from 3-arylsulfonylpropan-1-ol and subsequent one-pot cyclopropanation produced a regioisomer of the cyclopropyl sulfone.

The construction of functionalized cyclopropanes has received substantial attention, since these cyclopropanes serve as important intermediates for the versatile elaboration of four-,[1] five-,[2] seven-,[3] and eleven-membered rings[4] *via* vinylcyclopropane rearrangement and 1,4-dienes *via* solvolytic rearrangement.[5] The work of Zimmerman and Thyagarayan[6] and Truce and Lindy[7] on the reaction of 3-chloropropyl phenyl sulfone with a base as a novel preparation of cyclopropyl sulfones has stimulated considerable interest,[8–12] since the cyclopropanes can be converted into other functionalized derivatives by reductive cleavage of the sulfone moieties.[13] Although these functionalized cyclopropanes could be prepared from 1-methyl-3-phenylsulfonylpropyl toluene-*p*-sulfonate *via* conjugate addition of sodium benzenethiolate to methyl vinyl ketone[13] or from 3-bromoprop-1-enyl phenyl sulfone and alkyl Grignard reagents in the presence of catalytic amounts of cuprous salt,[14] or from γ,δ-epoxy sulfones by intramolecular nucleophilic substitutions developed by Gaoni,[15] there is still demand for a general and convenient procedure from readily available starting materials.

Results and Discussion

We have recently described efficient cyclopropanation of 1,3-bis(phenylthio)propanes,[16] 3-phenylthio-2-(phenylthiomethyl)propanamides,[17] and 3-phenylthio-3-(phenylthiomethyl)propanal.[18] As an extension of the reactions using sulfur-stabilized carbanions,[19] we now report a general and convenient synthetic method of cyclopropyl sulfone derivatives *via* alkylation of methyl phenyl sulfone and subsequent cyclization in a one-pot procedure.



Scheme 1 *Reagents and conditions:* i, BuLi (1.1 equiv.), THF, −78 °C, 0.5 h, 1,2-epoxyoctane (1.1 equiv.), −78 °C–room temp., H_2O; ii, (*a*) BuLi (1.1 equiv.), THF, −78 °C, 0.5 h; (*b*) epoxide **2** (1.1 equiv.), −78 °C–room temp.; (*c*) $PhSO_2Cl$ (1.0 equiv.), −78 °C–0 °C; (*d*) BuLi (1.1 equiv.), −78 °C–room temp.

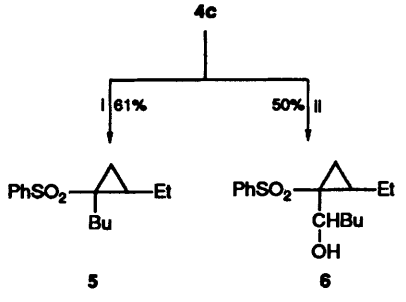
Lithiomethyl phenyl sulfone, derived from methyl phenyl sulfone **1** and butyllithium, readily reacted with 1,2-epoxyoctane in THF to give the lithium salt of 1-phenylsulfonylnonan-3-ol, which upon aqueous work-up gave 1-phenylsulfonylnonan-3-ol **3** (94% yield). When the lithium salt was treated with benzenesulfonyl chloride (1 equiv.) followed by butyllithium (1.1 equiv.), *trans*-2-hexylcyclopropyl phenyl sulfone **4a** was isolated in 84% overall yield from the sulfone **1**. Table 1 clearly indicates the generality of this one-pot cyclopropanation reaction. The stereochemical homogeneity was established by ^1H NMR spectroscopy and GLC. Thus, the ^1H NMR analysis of compound **4b** indicated only one methyl signal as a doublet (*J* 6 Hz) at δ 1.07 and GLC analysis showed a single peak. The 1-H of compound **4b** was split into a doublet (*J* 8 Hz) of triplets (*J* 4 Hz), which is consistent with a *trans* configuration of the methyl and phenylsulfonyl groups on the cyclopropane ring.[15,20]

Table 1 One-pot synthesis of $PhSO_2\overline{CHCH_2CHR}$ **4** from $MeSO_2Ph$ **1** and $\overline{OCH_2CHR}$ **2**

2/R	4/R	Yield (%)
a $Me(CH_2)_5$	$(CH_2)_5Me$	84
b Me	Me	75
c Et	Et	72
d Bu^i	Bu^i	66
e Bu	Bu	71
f $Me(CH_2)_4$	$(CH_2)_4Me$	67
g $Me(CH_2)_7$	$(CH_2)_7Me$	81
h $Me(CH_2)_{11}$	$(CH_2)_{11}Me$	71
i Ph	Ph	75

[a] The ring opening reaction of epoxides by lithiomethyl phenyl sulfone was carried out at −78 °C to room temperature for 15 h. No reaction took place at −78 °C for 1 h.

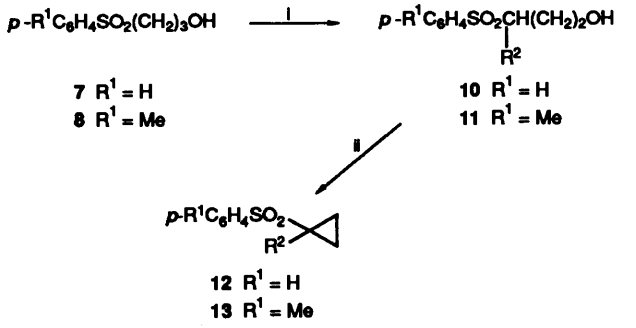
The utility of 2-alkylcyclopropyl phenyl sulfones is illustrated in Scheme 2. Thus, the lithiated cyclopropane **4c** can be alkylated to give the sulfone **5** in 61% yield or quenched with pentanal to give the alcohol **6** in 50% yield.



Scheme 2 *Reagents and conditions:* i, BuLi (1.1 equiv.), −78–0 °C, BuBr, −78–0 °C; ii, BuLi (1.1 equiv.), −78–0 °C, BuCHO, −78–0 °C

1-Alkyl-substituted cyclopropyl sulfones can be prepared by

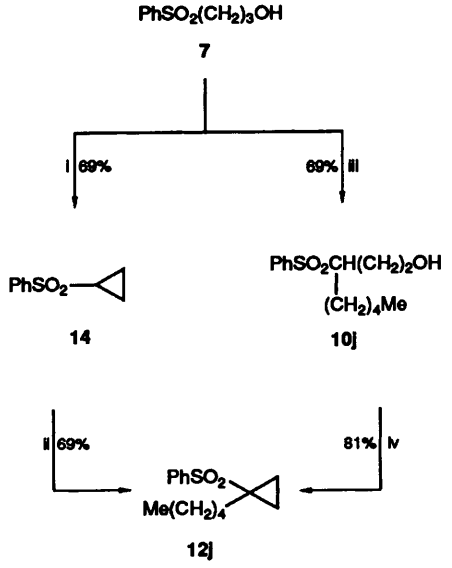
alkylation of dianions derived from 3-arylsulfonylpropan-1-ol **7** or **8** and subsequent one-pot cyclopropanation of the dianion (Scheme 3). Although Pine and co-workers reported that



Scheme 3 *Reagents and conditions:* i, BuLi (2.0 equiv.), THF–HMPA, R^2X **9** (1.1 equiv.); ii, (*a*) BuLi (1.1 equiv.), $PhSO_2Cl$ (1.0 equiv.); (*b*) BuLi (1.1 equiv.)

significant dialkylation is observed in the reaction of lithiomethyl phenyl sulfone and alkyl halides,[21] the alkylation of the dianions **7** and **8** led to exclusive formation of mono-substituted product.

It should be noted that the route to 1-alkylcyclopropyl phenyl sulfone **12j** *via* alkylation of 3-phenylsulfonylpropanol **7** and subsequent cyclization of **10j** provides a better yield compared to the formation of **12j** *via* cyclopropanation of **7** and alkylation of **14** as shown in Scheme 4.



Scheme 4 *Reagents and conditions:* i, BuLi (1.1 equiv.), $PhSO_2Cl$ (1.0 equiv.), BuLi (1.1 equiv.); ii, BuLi (1.1 equiv.), Me $(CH_2)_4Br$ (1.1 equiv.); iii, BuLi (2.0 equiv.), THF–HMPA, (1.1 equiv.), Me $(CH_2)_4Br$; iv, BuLi (1.1 equiv.), $PhSO_2Cl$ (1.0 equiv.), BuLi (1.1 equiv.)

In conclusion, the present method is operationally very simple, making use of readily available chemicals, and providing access to both the regioisomers of alkyl-substituted cyclopropyl phenyl sulfones.

Experimental

Tetrahydrofuran (THF) was distilled under argon from sodium benzophenone ketyl immediately before use. Hexamethyphosphoric triamide (HMPA) was distilled from calcium hydride and stored over Molecular Sieves 13X. Benzenesulfonyl chloride and epoxides were purified by distillation. IR spectra were recorded on a Hitachi Model 215 spectrophotometer. NMR spectra were obtained on a JEOL Model PS-100 (100 MHz) spectrometer in $CDCl_3$ with tetramethylsilane as an internal standard. *J* Values are given in Hz. GLC was performed on a Shimadzu Model GC-8A gas chromatograph using a 0.15 × 120 cm glass column (20% Silicone DC-550 on Celite 545). Thin layer chromatography was performed by using Merck precoated silica gel sheets 60F-254. Silica gel (Wakogel) of the size 100–200 mesh was used for column chromatography. Elemental analyses were performed by the Microanalytical Laboratory, operated by the Institue for Chemical Research, Kyoto University.

Preparation of 2-Alkylcyclopropyl Phenyl Sulfones: General Procedure.—To a stirred solution of methyl phenyl sulfone (1.56 g, 10.0 mmol) in THF (50 cm^3) at −78 °C was added dropwise butyllithium (11.0 mmol) and the resulting solution was stirred for 0.5 h. A solution of epoxide (11.0 mmol) in THF (3 cm^3) was then added and the solution allowed to warm to room temperature during 24 h. After this, a solution of benzenesulfonyl chloride (1.77 g, 10.0 mmol) in THF (3 cm^3) was added dropwise at −78 °C and the mixture was stirred for 8 h at 0 °C. It was then recooled to −78 °C and butyllithium (11.0 mmol) was added. The resulting mixture was allowed to warm to room temperature during 24 h before the addition of aqueous NH_4Cl (10 cm^3). The product was extracted with ether (3 × 50 cm^3) and the combined extracts were dried and evaporated to give a crude oil. Chromatography of this gave 2-alkylcyclopropyl phenyl sulfone **4**.

2-Hexylcyclopropyl phenyl sulfone **4a**. (Found: C, 67.55; H, 8.2. $C_{15}H_{22}O_2S$ requires C, 67.63; H, 8.32%); ν(thin film)/cm^{-1} 1450, 1312, 1150 and 1092; δ_H 0.75–0.95 (m, 4 H), 1.10–1.78 (m, 12 H), 2.09–2.27 (m, 1 H), 7.36–7.62 (m, 3 H) and 7.79–7.92 (m, 2 H); $\delta_C(CDCl_3)$ 12.43 (q), 13.57 (t), 19.91 (d), 22.02 (t), 28.20 (t), 28.27 (t), 31.17 (t), 31.31 (t), 38.72 (d), 126.95 (d), 128.70 (d) and 132.75 (d).

2-Methylcyclopropyl phenyl sulfone **4b**. (Found: C, 61.3; H, 6.3. $C_{10}H_{12}O_2S$ requires C, 61.20; H, 6.11%); ν(thin film)/cm^{-1} 1454, 1314, 1155 and 1099; δ_H 0.71–0.93 (m, 1 H), 1.07 (d, *J* 6, 3 H), 1.31–1.53 (m, 1 H), 1.59–1.87 (m, 1 H), 2.11–2.53 (dt, *J* 8, 4, 1 H), 7.40–7.67 (m, 3 H), 7.80–7.96 (m, 2 H).

2-Ethylcyclopropyl phenyl sulfone **4c**. (Found: C, 62.8; H, 6.7. $C_{11}H_{14}O_2S$ requires C, 62.58; H, 6.90%); ν(thin film)/cm^{-1} 1450, 1310, 1154 and 1094; δ_H 0.75–0.99 (m, 4 H), 1.07–1.79 (m, 4 H), 2.09–2.30 (m, 1 H), 7.34–7.61 (m, 3 H) and 7.76–7.90 (m, 2 H); $\delta_C(CDCl_3)$ 11.96 (t), 12.19 (d), 21.28 (d), 24.09 (t), 38.17 (d), 126.65 (d), 128.47 (d), 132.57 (d) and 140.20 (s).

2-Isobutylcyclopropyl phenyl sulfone **4d**. (Found: C, 65.3; H, 7.7. $C_{13}H_{18}O_2S$ requires C, 65.51; H, 7.61%); ν(thin film)/cm^{-1} 1452, 1312, 1158 and 1102; δ_H 0.81 (d, *J* 6, 6 H), 0.78–0.95 (m, 1 H), 1.03–1.28 (m, 2 H), 1.33–1.84 (m, 3 H), 2.11–2.34 (m, 1 H), 7.50–7.67 (m, 3 H) and 7.81–7.96 (m, 2 H).

2-Butylcyclopropyl phenyl sulfone **4e**. (Found: C, 65.3; H, 7.8. $C_{13}H_{18}O_2S$ requires C, 65.51; H, 7.61%); ν(thin film)/cm^{-1} 1454, 1315, 1155 and 1096; δ_H 0.67–0.96 (m, 4 H), 1.07–1.91 (m, 8 H), 2.10–2.31 (m, 1 H), 7.35–7.63 (m, 3 H) and 7.75–7.94 (m, 2 H).

2-Pentylcyclopropyl phenyl sulfone **4f**. ν(thin film)/cm^{-1} 1453, 1315, 1161 and 1101; δ_H 0.69–0.98 (m, 4 H), 1.07–2.80 (m, 10 H), 2.12–2.33 (m, 1 H), 7.46–7.71 (m, 3 H) and 7.81–8.02 (m, 2 H).

2-Octylcyclopropyl phenyl sulfone **4g**. (Found: C, 69.2; H, 9.1. $C_{17}H_{26}O_2S$ requires C, 69.34; H, 8.90%); $\nu(CHCl_3)$/cm^{-1} 1461, 1323, 1166 and 1107; δ_H 0.74–0.96 (m, 4 H), 1.09–1.64 (m, 16 H), 2.09–2.29 (m, 1 H), 7.50–7.66 (m, 3 H) and 7.82–7.94 (m, 2 H).

2-Dodecylcyclopropyl phenyl sulfone **4h**. ν(thin film)/cm^{-1} 1479, 1320, 1165 and 1105; δ_H 0.77–0.98 (m, 4 H), 1.07–1.72 (m, 24 H), 2.09–2.28 (m, 1 H), 7.39–7.64 (m, 3 H) and 7.80–7.94 (m, 2 H).

Phenyl 2-phenylcyclopropyl sulfone **4i**. (Found: C, 69.9; H, 5.3.

Table 2 Preparation of 1-alkylcyclopropyl aryl sulfones **10** or **11** from 3-arylsulfonylpropanol **7** or **8** *via* alkylation and subsequent cyclization

R^1	R^2X	Alkylated product **10, 11**	Yield (%)	Cyclopropane **12**	Yield (%)
H	$Me(CH_2)_4Br$	$PhSO_2CH(CH_2)_2OH$ $(CH_2)_4Me$	69	$PhSO_2$ $Me(CH_2)_4$	81
	9j	**10j**		**12j**	
H	$Me(CH_2)_6Br$	$PhSO_2CH(CH_2)_2OH$ $(CH_2)_6Me$	72	$PhSO_2$ $Me(CH_2)_6$	74
	9k	**10k**		**12k**	
Me	$Me(CH_2)_7I$	p-$MeC_6H_4SO_2CH(CH_2)_2OH$ $(CH_2)_7Me$	66	p-$MeC_6H_4SO_2$ $Me(CH_2)_7$	69
	9l	**11l**		**13l**	
Me	$Me(CH_2)_7I$	p-$MeC_6H_4SO_2CH(CH_2)_2OH$ $(CH_2)_7Me$	59[a]		
	9l	**11l**			
H	$Me(CH_2)_{11}Br$	$PhSO_2CH(CH_2)_2OH$ $(CH_2)_{11}Me$	74	$PhSO_2$ $Me(CH_2)_{11}$	68
	9m	**10m**		**12m**	
H	$Me_2CH(CH_2)_2Br$	$PhSO_2CH(CH_2)_2OH$ $(CH_2)_2CHMe_2$	78	$PhSO_2$ $Me_2CH(CH_2)_2$	85
	9n	**10n**		**12n**	
H	$CH_2=CHCH_2Br$	$PhSO_2CH(CH_2)_2OH$ $CH_2CH=CH_2$	62	$PhSO_2$ $CH_2=CHCH_2$	73
	9o	**10o**		**12o**	

[a] HMPA was not used.

$C_{15}H_{14}O_2S$ requires C, 69.74; H, 5.46%); $\nu(CHCl_3)/cm^{-1}$ 1451, 1312, 1155 and 1096; δ_H 1.20–1.59 (m, 1 H), 1.69–2.00 (m, 1 H), 2.57–3.01 (m, 2 H), 6.93–7.07 (m, 2 H), 7.17–7.34 (m, 3 H), 7.42–7.66 (m, 3 H) and 7.83–7.99 (m, 2 H).

1-Butyl-2-ethylcyclopropyl Phenyl Sulfone **5**. To a stirred solution of compound **4c** (1.05 g, 5.0 mmol) in THF (30 cm^3) at −78 °C was added butyllithium (5.5 mmol) and the solution was stirred for 1 h at 0 °C. It was then cooled to −78 °C when 1-bromobutane (0.47 g, 5.5 mmol) in THF (3 cm^3) was added. The cooling bath was removed, and the mixture was stirred at room temperature for 15 h. Work-up and chromatography gave the title compound **5** (0.81 g, 61% yield) (Found: C, 67.9; H, 8.6. $C_{15}H_{22}O_2S$ requires C, 67.63; H, 8.33%); ν(thin film)/cm^{-1} 1456, 1317, 1160 and 1101; δ_H 0.50 2.03 (m, 17 H), 7.31–7.61 (m, 3 H) and 7.71–7.89 (m, 2 H).

1-(2-Ethyl-1-phenylsulfonylcyclopropyl)pentanol **6**.—This was prepared (50% yield) by a similar procedure (Found: C, 64.7; H, 8.3. $C_{16}H_{24}O_3S$ requires C, 64.83; H, 8.16%); ν(thin film)/cm^{-1} 3495, 1458, 1317, 1155 and 1105; δ_H 0.72–2.00 (m, 18 H), 2.80 (br s, 1 H), 3.35–3.70 (m, 1 H), 7.39–7.65 (m, 3 H) and 7.78–7.93 (m, 2 H).

Preparation of 1-Alkylcyclopropyl Phenyl Sulfones. General Procedure.—To a stirred solution of 3-arylsulfonylpropan-1-ol **7** or **8** (10.0 mmol) in THF (30 cm^3) containing HMPA (3.0 cm^3) at −78 °C was added butyllithium (22.0 mmol) and the resulting solution was stirred for 30 min. Alkyl halide (11.0 mmol) was then added and the reaction mixture was stirred at room temperature for 15 h; it was then quenched with aqueous NH_4Cl (10 cm^3). The product was extracted with ether (3 × 50 cm^3) and the combined extracts were dried and evaporated. Chromatography gave 3-alkyl-3-arylsulfonylpropan-1-ol **10** or **11**. To a solution of compound **10** or **11** (7.0 mmol) in THF (30 cm^3) at −78 °C was added butyllithium (7.7 mmol) and the mixture was stirred for 30 min. After addition of benzenesulfonyl chloride (7.0 mmol) at −78 °C, the mixture was warmed to 0 °C and stirred for 8 h; butyllithium (7.7 mmol) was then added dropwise at this temperature. The resulting solution was allowed to warm to room temperature during 24 h before the addition of aqueous NH_4Cl (10 cm^3). The product was extracted with ether (3 × 50 cm^3) and the combined extracts were dried and evaporated to give a crude oil. Chromatography of this gave 1-alkylcyclopropyl aryl sulfone **12** or **13**.

1-Pentylcyclopropyl phenyl sulfone **12j**. (Found: C, 66.4; H, 8.2. $C_{14}H_{20}O_2S$ requires C, 66.63; H, 7.99%); ν(thin film)/cm^{-1} 1445, 1310, 1158 and 1095; δ_H 0.60–0.91 (m, 5 H), 0.98–1.35 (m, 6 H), 1.42–1.67 (m, 4 H), 7.32–7.57 (m, 3 H) and 7.71–7.86 (m, 2 H).

1-Heptylcyclopropyl phenyl sulfone **12k**. (Found: C, 68.35; H, 8.9. $C_{16}H_{24}O_2S$ requires C, 68.53; H, 8.63%); ν(thin film)/cm^{-1} 1452, 1313, 1158 and 1099; δ_H 0.72–0.95 (m, 5 H), 1.05–1.38 (m, 10 H), 1.46–1.74 (m, 4 H), 7.46–7.69 (m, 3 H), 7.85–7.99 (m, 2 H).

1-Octylcyclopropyl p-tolyl sulfone **13l**. (Found: C, 69.95; H, 8.95. $C_{18}H_{28}O_2S$ requires C, 70.08; H, 9.15%); ν(thin film)/cm^{-1} 1472, 1309, 1155 and 1099; δ_H 0.78–0.97 (m, 5 H), 1.00–1.29 (m, 12 H), 1.45–1.78 (m, 4 H), 2.43 (s, 3 H) and 7.25–7.95 (m, 4 H).

1-Dodecylcyclopropyl phenyl sulfone **12m**. (Found: C, 71.7; H, 9.6. $C_{21}H_{36}O_2S$ requires C, 71.95; H, 9.78%); $\nu(CHCl_3)/cm^{-1}$ 1452, 1312, 1225, 1155 and 1098; δ_H 0.77–0.98 (m, 5 H), 1.10–1.42 (m, 20 H), 1.50–1.72 (m, 4 H), 7.33–7.66 (m, 3 H) and 7.80–7.94 (m, 2 H).

1-(3-*Methylbutyl*)*cyclopropyl phenyl sulfone* **12n**. (Found: C, 66.3; H, 8.3. $C_{14}H_{20}O_2S$ requires C, 66.63; H, 7.99%); ν(thin film)/cm^{-1} 1450, 1313, 1157 and 1100; δ_H 0.76 (d, *J* 6, 6 H), 0.66–0.93 (m, 2 H), 1.07–1.46 (m, 3 H), 1.48–1.71 (m, 4 H), 7.38–7.62 (m, 3 H) and 7.75–7.90 (m, 2 H).

1-*Allylcyclopropyl phenyl sulfone* **12o**. (Found: C, 64.05; H, 6.95. $C_{12}H_{16}O_2S$ requires C, 64.25; H, 7.19%); ν(thin film)/cm^{-1} 1643, 1453, 1311, 1152 and 1095; δ_H 0.80–0.98 (m, 2 H), 1.47–1.65 (m, 2 H), 2.45 (d, *J* 7, 2 H), 4.81–5.07 (m, 2 H), 5.26–5.75 (m, 1 H), 7.46–7.72 (m, 3 H), 7.85–8.00 (m, 2 H).

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