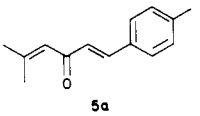
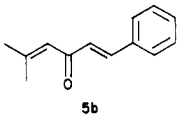
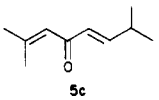
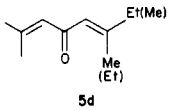
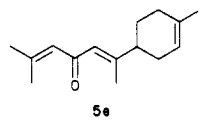
Table II. α,α′-Dienones 5a–e from the Reactions of 1 and Carbonyl Compounds 4a–e

carbonyl compound	product	base/solvent[a]	reactn temp, °C	yield,[b] %	config[c]
4a	 5a	NaH/DME EtONa/EtOH	70 78	54 40	*E*
4b	 5b	NaH/DME EtONa/EtOH	70 78	69 46	*E*
4c	 5c	NaH/DME	70	73	*E*
4d	 5d	NaH/DME	70	69	*E*/*Z* = 60/40
4e	 5e	NaH/DME	70	52	*E*

[a] All reactions were carried out under nitrogen atmosphere for 3 h. [b] Isolated yields and based on phosphonate 1. [c] Determined by ^{1}H NMR.

tolualdenyde (0.58 g, 4.83 mmol) in 2 mL of DME was added. The reaction mixture was heated with stirring at 70 °C for 3 h. The solvent was removed and the residue was dissolved in ether. After being washed with brine and drying over Na_2SO_4, the solvent was evaporated. Kugelrohr distillation at 150 °C (1 mmHg) gave the dienone **5a** (0.52 g, 54% yield) as pale yellow crystal: mp 72–73 °C (lit. mp 73.5–75 °C);[9] ^{1}H NMR δ 1.91, 2.16 (s, 6 H, $(CH_3)_2C$=CHCO), 2.33 (s, 3 H, *p*-$CH_3C_6H_4$), 6.21 (m, 1 H, $(CH_3)_2C$=CHCO), 6.61 (d, 1 H, COCH=CHAr, *J* = 16.5 Hz), 7.10 (d, 2 H, aromatic protons, *J* = 8.25 Hz), 7.40 (d, 2 H, aromatic protons, *J* = 8.25 Hz), 7.46 (d, 1 H, COCH=CHAr, *J* = 16.5 Hz); MS, *m*/*e* 200 (M^+). Anal. Calcd for $C_{14}H_{16}O$: C, 83.96 H, 8.05. Found: C, 84.20; H, 8.05.

The following α,α′-dienones **5b–e** were obtained from the same process as described above. The yields are listed in Table II.

(*E*)-1-Phenyl-3-oxo-5-methyl-1,4-hexadiene (5b): bp 150 °C (oven temperature) at 1 mmHg; ^{1}H NMR δ 1.80, 2.18 (s, 6 H, $(CH_3)_2C$=CHCO), 6.20 (m, 1 H, $(CH_3)_2CH$CO), 6.63 (d, 1 H, COCH=CHPh, *J* = 16.5 Hz), 7.41 (d, 1 H, COCH=CHPh, *J* = 16.5 Hz), 7.15–7.55 (m, 5 H, aromatic protons); MS calcd for $C_{13}H_{14}O$, 186.10441 (found 186.10351).

2,7-Dimethyl-4-oxo-2,5-octadiene (5c): bp 90 °C (oven temperature) at 1 mmHg; ^{1}H NMR δ 1.08 (d, 6 H, $(CH_3)_2CH$, *J* = 7 Hz), 1.89, 2.09 (s, 6 H, $(CH_3)_2C$=CHCO), 2.36 (m, 1 H, $(CH_3)_2CH$, *J* = 7.5 Hz), 5.86 (d, 1 H, COCH=CHCH$(CH_3)_2$, *J* = 16.5 Hz), 6.02 (m, 1 H, $(CH_3)_2C$=CHCO), 6.58 (dd, 1 H, COCH=CHCH=CHCH$(CH_3)_2$, *J* = 7.5, 16.5 Hz); MS calcd for $C_{10}H_{16}O$; 152.12006 (found, 152.11746).

2,6-Dimethyl-4-oxo-2,5-octadiene (5d): bp 100 °C (oven temperature) at 1 mmHg; ^{1}H NMR δ 1.04 (t, 3 H, CH_3CH_2, *J* = 7.5 Hz), 1.84 (s, 4 H, $(CH_3)_2C$=CHCOCH=$CCH_3(CH_2CH_3)$, methyl trans to carbonyl, overlapping signals), 2.09 (s, 5 H, $(CH_3)_2C$=CHCOCH=$CCH_3(CH_2CH_3)$, methyl cis to carbonyl, overlapping signals), 1.90–3.05 (m, 2 H, CH_3CH_2, overlapping signals), 5.88 (m, 2 H, vinyl protons); MS calcd for $C_{10}H_{16}O$, 152.12011 (found 152.12061).

(±)-α-Atlantone (5e): bp 150 °C (oven temperature) at 1 mmHg; ^{1}H NMR δ 1.55–2.40 [m, 19 H, including δ 1.66 (br s, CH=$C(CH_3)CH_2$), 1.85 (br s, COCH=$C(CH_3)_2$, methyl trans to carbonyl), 2.13 (br s, $C(CH_3)$=CHCOCH=$C(CH_3)_2$, two methyl cis to carbonyl)], 5.34 (m, 1 H, CH=$C(CH_3)CH_2$), 6.01 (br s, 2 H, C=CHCOCH=C); MS, *m*/*e* 218 (M^+); $[\alpha]^{23}_D$ +3° (C 0.74, EtOH).

(±)-*ar*-Turmerone (6). To a suspension of magnesium turnings (0.12 g, 4.8 mmol) in 20 mL of ether was added methyl iodide (0.35 mL, 4.8 mmol) in 10 mL of ether, and the reaction mixture was refluxed for 30 min. After the addition of copper(I) chloride (0.04 g, 0.4 mmol) and the dienone **5a** (0.8 g, 4 mmol), the solution was refluxed for 50 min and treated with saturated ammonium chloride solution. The organic layer was dried over Na_2SO_4 and the removal of ether afforded **6** (0.85 g, 98% yield). This sample was found to be almost pure on the basis of ^{1}H NMR spectrum. Kugelrorh distillation resulted in a significant loss of material due to thermal lability (0.20 g): bp 85 °C (oven temperature) at 0.5 mmHg; ^{1}H NMR δ 1.24 (d, 3 H, CH_3CHAr, *J* = 6.0 Hz), 1.83 (s, 3 H, $(CH_3)_2C$=CHCO, methyl trans to carbonyl), 2.09 (s, 3 H, $(CH_3)_2C$=CHCO, methyl cis to carbonyl), 2.28 (s, 3 H, $CH_3C_6H_4$), 6.00 (br s, 1 H, vinyl proton), 7.09 (s, 4 H, aromatic protons); MS, *m*/*e* 216 (M^+); $[\alpha]^{30}_{700-350}$ 0° (*c* 0.44, EtOH).

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Registry No. 1, 95485-29-3; **2a**, 3350-78-5; **2b**, 638-10-8; **2c**, 61985-22-6; **4a**, 104-87-0; **4b**, 100-52-7; **4c**, 78-84-2; **4d**, 78-93-3; (±)-**4e**, 70286-20-3; (*E*)-**5a**, 73839-46-0; (*E*)-**5b**, 79629-17-7; (*E*)-**5c**, 95485-30-6; (*E*)-**5d**, 95485-31-7; (*E*)-(±)-**5e**, 56362-49-3; (±)-**6**, 38142-58-4; dimethyl methanephosphonate, 756-79-6; (*Z*)-**5d**, 95485-32-8.

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Synthesis of Sulfones by Phase-Transfer Alkylation of Arenesulfinate Salts

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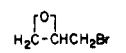
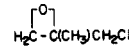
The sulfone group has enjoyed considerable recent popularity in organic synthesis as an activating function which can subsequently be removed under mild conditions.[1] One important method for the preparation of

Table I. *p*-Tolyl Sulfones from Alkyl Halides 1

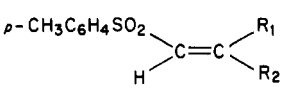
alkyl halide (1)	reactn time, h	sulfone yield, %	mp, °C (lit. mp)	spectroscopic data: ^{1}H NMR ($CDCl_3$) [MHz] δ (H, *J*/Hz); IR ($CHCl_3$) ν (cm^{-1})
$CH_3(CH_2)_5Cl$ (**1a**)	24	(0)		
$CH_3(CH_2)_6Br$ (**1b**)	8	**2b** (85)	50–51 (49–50)[9]	[220] 0.95 (t, 3), 1.25 (m, 8), 1.65 (m, 2), 2.45 (s, 3), 3.00 (m, 2), 7.5 (m, 4); 1300, 1150
CH_3CH_2I (**1c**)	6	**2c** (89)	50–51 (53–54)[10]	[60] 1.20 (t, 3, *J* = 7), 2.45 (s, 3), 3.07 (q, 2, *J* = 7), 7.5 (m, 4); 1300, 1150 (Nujol)
$CH_3(CH_2)_3CHBrCH_2CH_3$ (**1d**)	24	(0)		
$(CH_3)_2CHI$ (**1e**)	12	**2e** (65)	79–80 (77–79)[4]	[60] 1.30 (d, 6, *J* = 7), 2.45 (s, 3), 3.10 (sept, 1, *J* = 7), 7.5 (m, 4); 1300, 1150 (Nujol)
$C_6H_5CH_2Cl$ (**1f**)	8	**2f** (93)	142–143 (143–145)[4]	[60] 2.45 (s, 3), 4.25 (s, 2), 7.3 (m, 9); 1300, 1150
$CH_2{=}C(CH_3)CH_2Cl$ (**1g**)	12	**2g** (71)[a]	70.5–71.5	[220] 1.90 (m, 3), 2.45 (s, 3), 3.60 (s, 2), 4.60 (m, 1), 4.95 (m, 1), 7.5 (m, 4); 1340, 1160
$(CH_3)_2C{=}CHCH_2Br$ (**1h**)	12	**2h** (87)	77.5–79.5 (80–81)[11]	[60] 1.35 (s, 3), 1.75 (s, 3), 2.45 (s, 3), 3.80 (d, 2, *J*, = 7), 5.10 (t, 1, *J* = 7), 7.5 (m, 4); 1330, 1160
CH_3COCH_2Cl (**1i**)	6	**2i** (94)	50–51.5 (50–51)[12]	[60] 2.30 (s, 3), 2.45 (s, 3), 4.15 (s, 2), 7.5 (m, 4); 1725, 1340, 1160
$CH_3CHBrCO_2C_5H_5$ (**1j**)	8	**2j** (79)[b]	liquid	[220] 1.15 (t, 3, *J* = 7), 1.55 (d, 3, *J* = 7), 2.45 (s, 3), 4.00 (q, 1, *J* = 7), 4.10 (q, 2, *J* = 7), 7.5 (m, 4); 1750, 1340, 1140
CH_2I_2 (**1k**)	24	**2k** (55)	124–125 (128–129)[13]	[60] 2.45 (s, 3), 4.20 (s, 2), 7.5 (m, 4); 1300, 1150
$Br(CH_2)_3Cl$ (**1l**)	8	**2l** (93)	142–143 (144–145)[6]	[60] 2.23 (m, 2), 2.45 (s, 3), 3.20 (m, 2), 3.61 (m, 2); 1300, 1150
	12	**3a** (81)	120–121 (122)[7]	[90] 2.45 (s, 3), 2.55 (br s, 1), 4.35 (m, 2), 6.57 (d of t, 1, *J* = 15, 2), 7.00 (d, of t, 1, *J* = 15, 3), 7. 5 (m, 4); 3500, 1630, 1300, 1150
	12	**3b** (95%)[c]	105–107	[90] 2.00 (br s, 3), 2.45 (s, 3), 2.60 (br s, 1), 4.05 (br s, 2), 6.53 (m, 1), 7.5 (m, 4); 3500, 1630, 1300, 1180
		3c	95–96	[90] 1.95 (s, 3), 2.45 (s, 3), 3.5 (br s, 1), 4.55 (br s, 2), 6.15 (m, 1), 7.4 (m, 4)

[a] Anal. Calcd for $C_{11}H_{14}O_2S$: C, 62.83; H, 6.71; S, 15.25. Found: C, 62.8; H, 6.6% S, 15.2. [b] Purified by column chromatography. Anal. Calcd for $C_{12}H_{16}O_4S$: C, 56.23; H, 6.29; S, 12.51. Found: C, 56.0; H, 6.4, S, 12.2. [c] A 65:35 mixture separated by preparative TLC. Anal. Calcd for $C_{11}H_{14}O_3S$: C, 58.43; H, 6.19; S, 14.16. Found: C, 58.5; H, 6.1; S, 14.2.

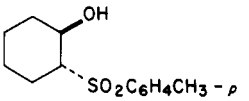
sulfones involves alkylation of sulfinate salts with alkyl halides, but traditional reaction conditions[1] using alcohols or dipolar aprotic solvents in which the two reactants have at least some solubility are often inconvenient. The use of a preformed tetra-*n*-butylammonium *p*-toluenesulfinate salt[2] or the benzenesulfinate form of a polystyrene anionic exchange resin[3] allows the reaction to be performed rapidly in typical nonpolar solvents, but the reagent must be prepared in a separate step. More recently a solid–liquid phase-transfer process was reported[4] which uses readily available sodium *p*-toluenesulfinate[5] as a solid in 1,2-dimethoxyethane in the presence of a small amount of tetra-*n*-butylammonium bromide. In this report we describe a complementary liquid–liquid phase-transfer method which consists of stirring alkyl halide, 1.5 equiv of sodium *p*-toluenesulfinate and a catalytic amount of a tetra-*n*-butylammonium salt (bromide or iodide) in a 4:3:3 mixture of water, benzene, and acetone at reflux. This two-phase solvent system gives cleaner, faster conversions than water–benzene alone. Little, if any, product was formed in the absence of the phase-transfer catalyst in cases where this was checked.

$$\underset{1}{R{-}X} + ArSO_2^- \rightarrow \underset{2}{RSO_2Ar} + X^-$$

Several different types of alkyl halides 1 were converted to the corresponding sulfones (**2**, Ar = *p*-$CH_3C_6H_4$) in good yields as shown in Table I. However, this procedure appears to be limited to primary bromides and iodides and secondary iodides. Activated chlorides and bromides (allylic, benzyl, and α-carbonyl) are also satisfactory starting materials. Methylene iodide and 1-bromo-3-chloro-propane gave monohalo sulfones which are useful synthetic intermediates. For example, 3-chloropropyl *p*-tolyl sulfone was readily converted to cyclopropyl *p*-tolyl sulfone[6] by a phase-transfer reaction with aqueous sodium hydroxide. 1-Bromo-2,3-epoxypropane gave (*E*)-3-[(*p*-methylphenyl)sulfonyl]-2-propen-1-ol (**3a**) as the major



3



4

a, R_1 = H, R_2 = CH_2OH;
b, R_1 = CH_3, R_2 = CH_2OH;
c, R_1 = CH_2OH; R_2 = CH_3

product.[7] In this case the intermediate epoxy sulfone suffers β-elimination under the reaction conditions.[7,8] Analogously, 1-chloro-2,3-epoxy-2-methylpropane was converted to a 65:35 mixture of (*E*)- and (*Z*)-3-[(*p*-methylphenyl)sulfonyl]-2-methyl-2-propen-1-ol (**3b** and **3c**). (The *E* structure was assigned to the isomer whose olefinic hydrogen was similar in chemical shift to that of the corresponding hydrogen of **3a**.) Finally, epoxycyclohexane gave *trans*-2-[(*p*-methylphenyl)sulfonyl]cyclohexanol (**4**) indicating that this procedure will also be useful in transforming epoxides into β-hydroxy sulfones.[7]

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Experimental Section

General Procedure for the Preparation of Sulfones. A mixture of 17 g (0.09 mol) of sodium *p*-toluenesulfinate,[5] 0.06 mol of alkyl halide or epoxide, and 1.5 g of the phase-transfer catalyst (tetra-*n*-butylammonium bromide or iodide) in a solvent system consisting of 20 mL of water, 15 mL of benzene, and 15 mL of acetone was placed in a flask equipped with a mechanical stirrer and a reflux condenser. Upon heating to 80–85 °C a two-phase liquid mixture was obtained. The course of reaction was generally monitored by following the disappearance of organic halide by GC or TLC. After the indicated period, the reaction mixture was poured into a separatory funnel containing 50 mL of water and 50 mL of ether. The layers were separated, the aqueous layer was extracted again with ether, and the combined extracts were dried ($MgSO_4$) and concentrated. The resulting viscous oil usually crystallized on standing or upon the addition of a small amount of hexane with cooling. The crude product was collected and recrystallized from ethanol.

Cyclopropyl *p*-Tolyl Sulfone. A mixture of 1.63 g of 3-chloropropyl *p*-tolyl sulfone, 15 g of NaOH, 1.5 g of tetra-*n*-butylammonium iodide, 15 mL of water, 8 mL of benzene, and 8 mL of acetone was placed in a flask equipped with a mechanical stirrer and a reflux condenser. A two-phase mixture was obtained upon heating at 80 °C. After 18 h the reaction mixture was poured into a mixture of water and ether. The organic layer was separated, washed with dilute hydrochloric acid, and dried ($MgSO_4$). Removal of the solvent gave 1.3 g of an oil. Distillation under vacuum gave 0.82 g (61%) of cyclopropyl *p*-tolyl sulfone, which readily crystallized: bp 175–178 °C (0.05 torr), mp 65–66 °C (lit.[6] mp 65–66 °C); IR (CCl_4) 1300, 1150 cm^{-1}; NMR (220 MHz, CCl_4) δ 1.04 (m, 2), 1.36 (m, 2), 2.46 (m, 1), 2.45 (s, 3), 7.45 (m, 4).

***trans*-2-[(*p*-Methylphenyl)sulfonyl]cyclohexanol (4).** The reaction of 5.9 g of cyclohexene oxide by the general procedure required 20 h. Recrystallization from 1:1 acetone–water gave 12.3 g (89%) of **4**: mp 120–121 °C (lit.[14] mp 123 °C); IR 3500, 1260, 1160 cm^{-1}; NMR (90 MHz) δ 1.2–2.0 (m, 8), 2.45 (s, 3), 2.95 (m, 1), 3.90 (m, 1), 4.4 (br s, 1), 7.45 (m, 4).

Registry No. **1a**, 544-10-5; **1b**, 629-04-9; **1c**, 75-03-6; **1d**, 1974-05-6; **1e**, 75-30-9; **1f**, 100-44-7; **1g**, 563-47-3; **1h**, 870-63-3; **1i**, 78-95-5; **1j**, 535-11-5; **1k**, 75-11-6; **1l**, 109-70-6; **2b**, 95314-81-1; **2c**, 7569-34-8; **2e**, 51751-71-4; **2f**, 5395-20-0; **2g**, 16192-04-4; **2h**, 15543-64-3; **2i**, 5366-49-4; **2j**, 95314-82-2; **2k**, 37891-96-6; **2l**, 19432-95-2; **3a**, 95314-83-3; **3b**, 95314-84-4; **3c**, 95314-85-5; **4**, 95314-86-6; 2-(chloromethyl)-2-methyloxirane, 598-09-4; 2-(bromomethyl)oxirane, 3132-64-7; cyclopropyl *p*-tolyl sulfone, 91061-30-2; sodium *p*-toluenesulfinate, 824-79-3; cyclohexene oxide, 286-20-4.

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Hindered Amines.[1] Hindered Monoazacrown Ethers

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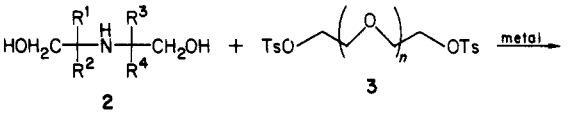
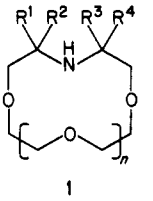
There has been wide interest in the chemistry of hindered amines[2] and azacrown ethers.[3] The hindered azacrown ethers **1** contain both of these structural features and might offer many other unique applications. For example, the potassium salts of **1** could provide nonassociated, nonnucleophilic bases in elimination reactions.[4] The stable nitroxyl radicals of **1** may be the ideal probe for studying dynamic processes in micellar and related membrane-like systems.[5] In this note we would like to report a simple synthesis of the hindered azacrown ethers **1**.

Bis(1,1-dialkyl-2-hydroxyethyl)amine **2** can be prepared in large quantities from very inexpensive raw materials according to our recent report.[6] When the metal salts of **2** were alkylated with the ditosylates of oligomeric ethylene glycols **3**;[7] **1** were obtained in reasonable yields.



2 **3**



1

a, R^1, R^2, R^3, R^4 = Me; n = 2
b, R^1, R^2, R^3, R^4 = Me; n = 3
c, R^1, R^2, R^3 = Me; R^4 = Et; n = 2
d, R^1, R^2 = Me, R^3, R^4 = $-(CH_2)_5-$; n = 2
e, R^1, R^2, R^3, R^4 = Me; n = 1

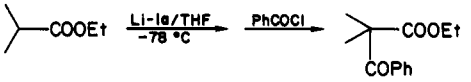
Table I

1[a]	template metal (M)	yield,[b] %	bp, °C/mmHg
a	Na	60	88–91/0.04
b	K	41	101–2/0.05
c	Na	55	92–6/0.05
d	Na	49	132–5/0.07
e	Li	7	68–9/0.20 (mp 39–42 °C)

[a] All compounds have acceptable microanalyses: C, ±0.29; H, ±0.20; N, ±0.18. [b] Yield of pure product.

The nitroxyl radical of **1a** or **1b** (4×10^{-4} M in methanol) shows a typical triplet with α_n = 15.5 G in its ESR spectra. The splitting constant does not change[5] when the same methanol solution is saturated with either NaSCN or KSCN.

The lithium amide from **1a** (Li-**1a**) can deprotonate[8] ethyl isobutyrate in THF at −78 °C; reaction with benzoyl chloride then gives ethyl α-benzoylisobutyrate in >80% yield.



Experimental Section

^1H NMR spectra were recorded on a Bruker WH-200 spectrometer. $CDCl_3$ was used as solvent with Me_4Si added as internal standard. Mass spectra were obtained on a Varian MAT 311A spectrometer. Microanalyses were performed by Huffman Lab, in Wheatridge, CO.

General Procedure for Preparing 1. Metal (0.03 mol) was added in small portions to a solution of **2** (0.01 mol) in 85 mL of *tert*-butyl alcohol, and the mixture was slowly warmed to 60

(1) Part 8 of a series. For part 7, see: Lai, J. T. *Synthesis* **1984**, 124.
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