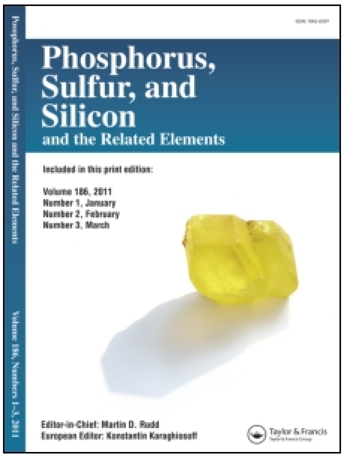

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Selective Oxidation of Sulfides to Sulfoxides and Sulfones Using <i>n</i>-Butyltriphenylphosphonium Dichromate (Bu$^{<i>n</i>}$PPh$_3$)$_2$Cr$_2$O$_7$ in the Presence of Aluminium Chloride in Solution and Under Microwave Irradiation

Iraj Mohammadpoor-Baltork[a]; Hamid Reza Memarian[a]; Kiumars Bahrami[a]; Karim Esmayilpour[a]
[a] Department of Chemistry, Isfahan University, Isfahan, Iran

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Selective Oxidation of Sulfides to Sulfoxides and Sulfones Using *n*-Butyltriphenylphosphonium Dichromate $(Bu^nPPh_3)_2Cr_2O_7$ in the Presence of Aluminium Chloride in Solution and Under Microwave Irradiation

Iraj Mohammadpoor-Baltork
Hamid Reza Memarian
Kiumars Bahrami
Karim Esmayilpour
Department of Chemistry, Isfahan University, Isfahan, Iran

A wide variety of sulfides are efficiently oxidized to their corresponding sulfoxides and sulfones in excellent yields using n-butyltriphenylphosphonium dichromate (BTPPDC) in the presence of aluminium chloride in acetonitrile solution and under microwave irradiation. In addition, selective oxidation of sulfides in the presence of functional groups such as a carbon–carbon double bond, ketone, oxime, aldehyde, ether, and acetal can be considered as a noteworthy advantage of this method.

Keywords Dichromate; oxidation; sulfides; sulfones; sulfoxides

INTRODUCTION

The selective oxidation of sulfides to sulfoxides or sulfones is an important synthetic transformation. These compounds are used as important intermediates in organic synthesis.[1] The most widely used method for the preparation of sulfoxides and sulfones is the oxidation of the corresponding sulfides. A number of methods have been reported for these transformations in the literature.[2–29] However, some of these methods show limitations such as long reaction times, low yields of the products, expensive reagents, and poor selectivity. On the other hand, selective oxidation of sulfides to sulfoxides and sulfides to sulfones using the same reagent system under adjusted reaction conditions is of practical

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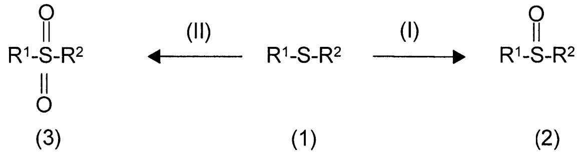
Address correspondence to Iraj Mohammadpoor-Baltork, Isfahan University, Department of Chemistry, Isfahan 81746-73441, Iran. E-mail: imbaltork@sci.ui.ac.ir

importance and only a few reports are available in the literature for this purpose.[30] Therefore, there is still a need for a simple and less expensive method for the conversion of sulfides to sulfoxides and sulfones.

In recent years organic reactions assisted by microwave irradiation have gained special attention.[31] The chief features of the microwave reactions are the much-improved reaction rates, formation of cleaner products, and operational simplicity.

RESULTS AND DISCUSSION

In this article, we describe a successful application of *n*-butyltriphenylphosphonium dichromate/$AlCl_3$ system for the selective oxidation of sulfides to sulfoxides and sulfides to sulfones according to the appropriate adjustment of the reaction conditions in solution and under microwave irradiation (Scheme 1).

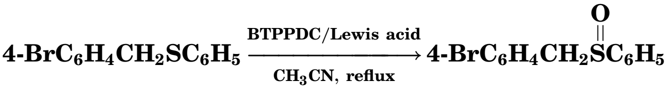


(I): BTPPDC (1.2–1.5 eq.), $AlCl_3$ (1 eq.), CH_3CN, reflux or MW
(II): BTPPDC (1.5–3 eq.), $AlCl_3$ (1 eq.), CH_3CN, reflux or MW

SCHEME 1

BTPPDC is an inexpensive reagent and easily is prepared from the commercially available starting materials. Oxidation of different organic compounds using this reagent previously has been reported.[32] At first, the oxidation of 4-bromobenzyl phenyl sulfide (**1e**) as a model compound was examined with BTPPDC in refluxing acetonitrile. Under this condition, the reaction did not proceed at all after 2 h and the starting material was isolated intact from the reaction mixture. In recent years the effect of Lewis acids as a promoter or catalyst for the oxidation of organic substrates has been reported.[33] Therefore, the effect of several Lewis acids upon the reactivity of BTPPDC for the oxidation of 4-bromobenzyl phenyl sulfide in refluxing acetonitrile has been studied. The results show that only $AlCl_3$ is effective for this purpose (Table I).

The oxidation of 4-bromobenzyl phenyl sulfide to the corresponding sulfoxide also was investigated with BTPPDC/$AlCl_3$ in various solvents. Among the solvents that were examined, acetonitrile turned out to be a suitable solvent for this transformation (Table II).

TABLE I


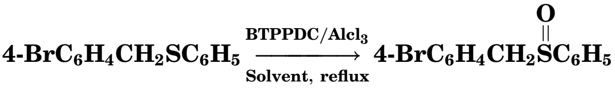
$$4\text{-BrC}_6\text{H}_4\text{CH}_2\text{SC}_6\text{H}_5 \xrightarrow[\text{CH}_3\text{CN, reflux}]{\text{BTPPDC/Lewis acid}} 4\text{-BrC}_6\text{H}_4\text{CH}_2\overset{\overset{\text{O}}{\|}}{\text{S}}\text{C}_6\text{H}_5$$

Entry	Lewis acid[a]	Time (min)	Yield (%)[b]
1	$AlCl_3$	70	94
2	$BiCl_3$	70	76
3	$SnCl_2.2H_2O$	70	75
4	$NiCl_2.6H_2O$	70	60
5	$FeCl_3.6H_2O$	70	70
6	$CoCl_2.6H_2O$	70	70

[a] Sulfide: BTPPDC: Lewis acid = 1:1.5:1.
[b] Isolated yields.

The optimum molar ratio of sulfide to BTPPDC to $AlCl_3$ (1:1.5:1) is found to be ideal for the complete conversion of sulfides to sulfoxides in solution and 1:1.2:1 under microwave irradiation, while with lesser amounts (for example, 1:1:0.75 and 1:1:1) the reaction remains incomplete. As shown in Table III, a series of diaryl, dibenzyl, aryl benzyl, dialkyl, and cyclic sulfides **1a–s** were then reacted with this reagent system in refluxing acetonitrile, which afforded the corresponding sulfoxides **2a–s** in 85–95% yields within 40–240 min. The oxidation of these sulfides to sulfoxides also was investigated under microwave irradiation. Under these conditions, the corresponding sulfoxides were obtained in 85–96% yields and in a very short reaction time (0.75–5.5 min), (Table III). However, in oxidation of alkyl aryl sulfides such as methyl phenyl sulfide and 4-chlorophenyl methyl sulfide, a mixture of sulfoxide (~65%) and sulfone (~30%) was isolated from the reaction mixture.

In order to extend the scope of this reagent system further, the oxidation of sulfides to their corresponding sulfones also was investigated in

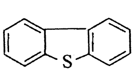
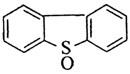
TABLE II


$$4\text{-BrC}_6\text{H}_4\text{CH}_2\text{SC}_6\text{H}_5 \xrightarrow[\text{Solvent, reflux}]{\text{BTPPDC/Alcl}_3} 4\text{-BrC}_6\text{H}_4\text{CH}_2\overset{\overset{\text{O}}{\|}}{\text{S}}\text{C}_6\text{H}_5$$

Entry	Solvent[a]	Time (min)	Yield (%)[b]
1	CH_3CN	70	94
2	CH_3COCH_3	70	81
3	$CHCl_3$	70	60
4	CH_2Cl_2	70	50
5	*n*-Hexane	70	10

[a] Sulfide: BTPPDC: $AlCl_3$ = 1:1.5:1.
[b] Isolated yields.

TABLE III Oxidation of Sulfides to Sulfoxides Using $(Bu^nPPh_3)_2Cr_2O_7/AlCl_3$

		Time (min)		Yield (%)[c]	
Sulfide (**1**)	Sulfoxide (**2**)	Solution[a]	MW[b]	Solution	MW
$(C_6H_5)_2S$ (**1a**)	$(C_6H_5)_2SO$ (**2a**)	200	5	92	92
$C_6H_5CH_2SC_6H_5$ (**1b**)	$C_6H_5CH_2SOC_6H_5$ (**2b**)	90	2	93	94
$(C_6H_5CH_2)_2S$ (**1c**)	$(C_6H_5CH_2)_2SO$ (**2c**)	60	1	93	95
4-$MeC_6H_4CH_2SC_6H_5$ (**1d**)	4-$MeC_6H_4CH_2SOC_6H_5$ (**2d**)	90	2	93	94
4-$BrC_6H_4CH_2SC_6H_5$ (**1e**)	4-$BrC_6H_4CH_2SOC_6H_5$ (**2e**)	70	1.25	94	96
4-$O_2NC_6H_4CH_2SC_6H_5$ (**1f**)	4-$O_2NC_6H_4CH_2SOC_6H_5$ (**2f**)	55	1	95	96
4-$MeC_6H_4SCH_2C_6H_5$ (**1g**)	4-$MeC_6H_4SOCH_2C_6H_5$ (**2g**)	60	1	94	95
4-$BrC_6H_4SCH_2C_6H_5$ (**1h**)	4-$BrC_6H_4SOCH_2C_6H_5$ (**2h**)	115	2.5	95	95
4-$ClC_6H_4SCH_2C_6H_5$ (**1i**)	4-$ClC_6H_4SOCH_2C_6H_5$ (**2i**)	180	4	93	94
4-$BrC_6H_4CH_2SCH_2C_6H_5$ (**1j**)	4-$BrC_6H_4CH_2SOCH_2C_6H_5$ (**2j**)	60	1	94	95
4-$BrC_6H_4CH_2SC_6H_4$4-Me (**1k**)	4-$BrC_6H_4CH_2SOC_6H_4$4-Me (**2k**)	70	1.25	93	95
4-$BrC_6H_4CH_2SC_6H_4$4-Br (**1l**)	4-$BrC_6H_4CH_2SOC_6H_4$4-Br (**2l**)	90	2	92	94
4-$BrC_6H_4CH_2SC_6H_4$4-Cl (**1m**)	4-$BrC_6H_4CH_2SOC_6H_4$4-Cl (**2m**)	120	2.5	95	94
4-$O_2NC_6H_4CH_2SC_6H_4$4-Cl (**1n**)	4-$O_2NC_6H_4CH_2SOC_6H_4$4-Cl (**2n**)[d]	100	2	85	86
2-$ClC_6H_4CH_2SC_6H_4$4-Cl (**1o**)	2-$ClC_6H_4CH_2SOC_6H_4$4-Cl (**2o**)	180	4	92	93
$(n\text{-}C_3H_7)_2S$ (**1p**)	$(n\text{-}C_3H_7)_2SO$ (**2p**)[d]	40	0.75	85	85
$(n\text{-}C_4H_9)_2S$ (**1q**)	$(n\text{-}C_4H_9)_2SO$ (**2q**)[d]	60	1	88	85
$n\text{-}C_8H_{17}SCH_2CH_2CHMe_2$ (**1r**)	$n\text{-}C_8H_{17}SOCH_2CH_2CHMe_2$ (**2r**)	220	5.25	93	94
 (**1s**)	 (**2s**)[d]	240	5.5	86	85

[a] Sulfide: BTPPDC: $AlCl_3$ = 1:1.5:1.

[b] Sulfide: BTPPDC: $AlCl_3$ = 1:1.5:1.

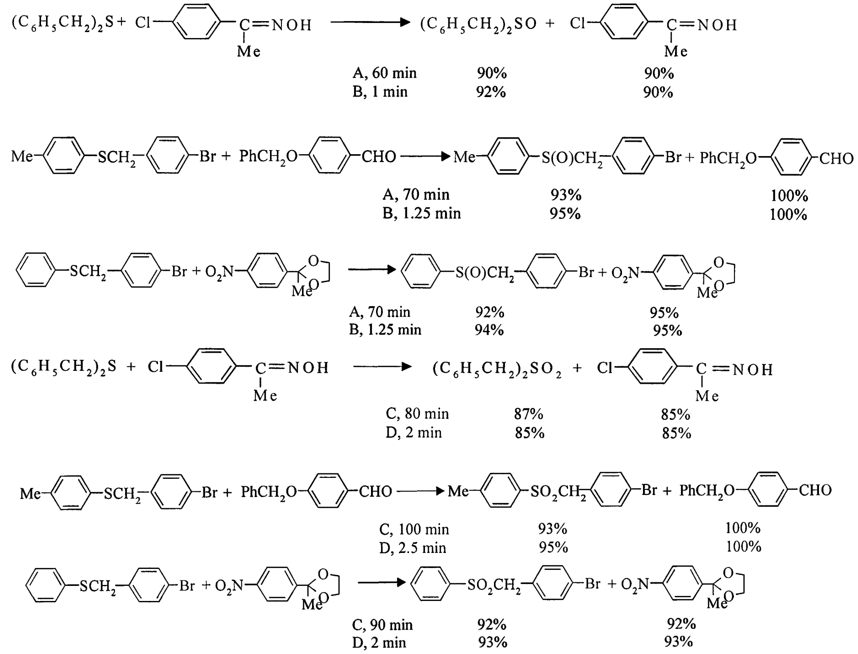
[c] Isolated yields.

[d] 3–10% of sulfone was obtained from the reaction mixture.

solution and under microwave irradiation. The optimum molar ratios of sulfide to BTPPDC to $AlCl_3$ in solution and under microwave irradiation were found to be 1:2–3:1 and 1:1.5–2.5:1, respectively. When a wide range of diaryl, dialkyl, dibenzyl, aryl benzyl, alkyl aryl, and cyclic sulfides **1a–z**, **1a′–d′** were treated with BTPPDC in the presence of $AlCl_3$ in refluxing acetonitrile, the corresponding sulfones **3a–z** and **3a′–d′** were obtained in 92–96% yields. The reaction time usually is between 30–270 min. Under microwave irradiation, these sulfides were oxidized to their corresponding sulfones in 93–98% yields with reaction periods ranging between 0.75–5.5 min (Table IV). Under microwave irradiation, acetonitrile was used for homogenization of the reaction mixture. The polar character of this solvent also seems to increase the reaction temperature, so the reaction is completed in a short time.[31c] The results show that the yields of the products are comparable in solution and under microwave irradiation, but the reaction times considerably are shorter under microwave irradiation (Tables III and IV). Also the molar ratio of BTPPDC to sulfide is lower under microwave irradiation.

It is noteworthy that sulfides containing functional groups such as alkene and ketone are oxidized selectively without any interference from these groups (Table IV, entries **1t**, **1c′**, and **1d′**). We also have monitored competitive oxidation of sulfides in the presence of oxime, aldehyde, ether, and acetal. The results are demonstrated in Scheme 2. These observations clearly show that the method is applicable for the selective oxidation of sulfides in the presence of the previously mentioned functional groups and can be considered as a useful practical achievement in oxidtion of sulfides to sulfoxides and sulfones.

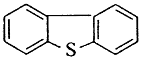
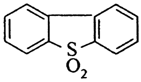
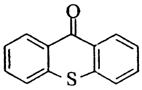
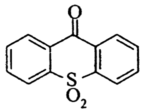
In Table V, the results of the oxidation of benzyl phenyl sulfide (**1b**) to benzyl phenyl sulfoxide (**2b**) and benzyl phenyl sulfone (**3b**) by our method are compared with those reported by other methods. As can be seen, this method is superior to some previously reported methods in terms of yields, reaction times, and the amount of the reagent used for successful oxidation.



A = BTPPDC:substrates:$AlCl_3$ = 1.5:1:1:1, CH_3CN, reflux.
B = BTPPDC:substrates:$AlCl_3$ = 1.2:1:1:1, CH_3CN, MW.
C = BTPPDC:substrates:$AlCl_3$ = 3:1:1:1, CH_3CN, reflux.
D = BTPPDC:substrates:$AlCl_3$ = 2.5:1:1:1, CH_3CN, MW.

SCHEME 2

TABLE IV Oxidation of Sulfides to Sulfones Using $(Bu^nPPh_3)_2Cr_2O_7/AlCl_3$

		Time (min)		Yield (%)^c	
Sulfide (**1**)	Sulfone (**3**)	Solution^a	MW^b	Solution	MW
$(C_6H_5)_2S$ (**1a**)	$(C_6H_5)_2SO_2$ (**3a**)	180	4.5	95	96
$C_6H_5CH_2SC_6H_5$ (**1b**)	$C_6H_5CH_2SO_2C_6H_5$ (**3b**)	100	2.5	96	97
$(C_6H_5CH_2)_2S$ (**1c**)	$(C_6H_5CH_2)_2SO_2$ (**3c**)	80	2	95	96
4-$MeC_6H_4CH_2SC_6H_5$ (**1d**)	4-$MeC_6H_4CH_2SO_2C_6H_5$ (**3d**)	65	1	96	98
4-$BrC_6H_4CH_2SC_6H_5$ (**1e**)	4-$BrC_6H_4CH_2SO_2C_6H_5$ (**3e**)	90	2	93	96
4-$O_2NC_6H_4CH_2SC_6H_5$ (**1f**)	4-$O_2NC_6H_4CH_2SO_2C_6H_5$ (**3f**)	70	2	95	97
4-$MeC_6H_4SCH_2C_6H_5$ (**1g**)	4-$MeC_6H_4SO_2CH_2C_6H_5$ (**3g**)	100	2.5	93	93
4-$BrC_6H_4SCH_2C_6H_5$ (**1h**)	4-$BrC_6H_4SO_2CH_2C_6H_5$ (**3h**)	120	3	95	96
4-$ClC_6H_4SCH_2C_6H_5$ (**1i**)	4-$ClC_6H_4SO_2CH_2C_6H_5$ (**3i**)	150	3	93	93
4-$BrC_6H_4CH_2SCH_2C_6H_5$ (**1j**)	4-$BrC_6H_4CH_2SO_2CH_2C_6H_5$ (**3j**)	45	0.75	96	96
4-$BrC_6H_4CH_2SC_6H_4$4-Me (**1k**)	4-$BrC_6H_4CH_2SO_2C_6H_4$4-Me (**3k**)	100	2.5	93	95
4-$BrC_6H_4CH_2SC_6H_4$4-Br (**1l**)	4-$BrC_6H_4CH_2SO_2C_6H_4$4-Br (**3l**)	130	4	94	96
4-$BrC_6H_4CH_2SC_6H_4$4-Cl (**1m**)	4-$BrC_6H_4CH_2SO_2C_6H_4$4-Cl (**3m**)	125	3	94	97
4-$O_2NC_6H_4CH_2SC_6H_4$4-Cl (**1n**)	4-$O_2NC_6H_4CH_2SO_2C_6H_4$4-Cl (**3n**)	75	1.5	92	94
2-$ClC_6H_4CH_2SC_6H_4$4-Cl (**1o**)	2-$ClC_6H_4CH_2SO_2C_6H_4$4-Cl (**3o**)	180	4	95	96
$(n\text{-}C_3H_7)_2S$ (**1p**)	$(n\text{-}C_3H_7)_2SO_2$ (**3p**)	75	1.5	93	94
$(n\text{-}C_4H_9)_2S$ (**1q**)	$(n\text{-}C_4H_9)_2SO_2$ (**3q**)	130	3	96	95
$n\text{-}C_8H_{17}SCH_2CH_2CHMe_2$ (**1r**)	$n\text{-}C_8H_{17}SO_2CH_2CH_2CHMe_2$ (**3r**)	200	5	94	95
 **(1s)**	 **(3s)**	240	5	95	95
 **(1t)**	 **(3t)**	270	5.5	92	93
$n\text{-}C_8H_{17}SMe$ (**1u**)	$n\text{-}C_8H_{17}SO_2Me$ (**3u**)	90	2	95	94
$n\text{-}C_8H_{17}SCH_2CH_3$ (**1v**)	$n\text{-}C_8H_{17}SO_2CH_2CH_3$ (**3v**)	100	2.5	94	95
C_6H_5SMe (**1w**)	$C_6H_5SO_2Me$ (**3w**)	40	1	94	95
4-MeC_6H_4SMe (**1x**)	4-$MeC_6H_4SO_2Me$ (**3x**)	30	0.75	96	97
4-ClC_6H_4SMe (**1y**)	4-$ClC_6H_4SO_2Me$ (**3y**)	120	3	94	96
$C_6H_5S(n\text{-}C_3H_7)$ (**1z**)	$C_6H_4SO_2(n\text{-}C_3H_7)$ (**3z**)	75	1.5	96	97
$C_6H_5S(n\text{-}C_4H_9)$ (**1a′**)	$C_6H_5SO_2(n\text{-}C_4H_9)$ (**3a′**)	90	2	94	95
$C_6H_5SCHMe_2$ (**1b′**)	$C_6H_5SO_2CHMe_2$ (**3b′**)	120	3	93	94
$C_6H_5SCH_2CH{=}CH_2$ (**1c′**)	$C_6H_5SO_2CH_2CH{=}CH_2$ (**3c′**)	45	1	94	94
$(CH_2{=}CHCH_2)_2S$ (**1d′**)	$(CH_2{=}CHCH_2)_2SO_2$ (**3d′**)	40	0.75	95	95

^{a}Sulfide: BTPPDC: $AlCl_3$ = 1:3:1 and 1:2:1 for entries 1a–1v and 1w–1d′, respectively.

^{b}Sulfide: BTPPDC: $AlCl_3$ = 1:2.5:1 and 1:1.5:1 for entries 1a–1v and 1w–1d′, respectively.

^{c}Isolated yield.

In conclusion the results presented in this article demonstrate that BTPPDC/$AlCl_3$ can be used for the efficient and selective oxidation of sulfides to sulfoxides and sulfones. Short reaction times, excellent yields, easy workup, easy preparation, and low cost of the reagent are

TABLE V Comparison of Oxidation of Benzyl Phenyl Sulfide (1b) to Benzyl Phenyl Sulfoxide (2b) and Benzyl Phenyl Sulfone (3b) by BTPPDC/AlCl$_3$ with Some of Those Reported in the Literature

Reagen (oxidant/substrate)	Product	Yield % (Time, min)
BTPPDC/AlCl$_3$/CH$_3$CN/reflux (1.5:1)	**2b**	93 (90)
BTPPDC/AlCl$_3$/MW (1.2:1)	**2b**	94 (2)
NaIO$_4$/wet SiO$_2$/MW (1.7:1)[30f]	**2b**	83 (2.5)
PhCH$_2$PPh$_3$HSO$_5$/CH$_3$CN/reflux (1.5:1)[18b]	**2b**	88 (12 h)
Ba(MnO$_4$)$_2$/CH$_3$CN/reflux (6:1)[21a]	**2b**	88 (4 h)
CAN/wet SiO$_2$/CH$_2$Cl$_2$/rt (2:1)[8]	**2b**	96 (45)
BTPPDC/AlCl$_3$/CH$_3$CN/reflux (3:1)	**3b**	96 (100)
BTPPDC/AlCl$_3$/MW (2.5:1)	**3b**	97 (2.5)
NaIO$_4$/wet SiO$_2$/MW (3:1)[30f]	**3b**	87 (2.5)
MMPP/wet SiO$_2$/CH$_2$Cl$_2$/reflux (3:1)[27]	**3b**	94 (75)
Oxone/AlCl$_3$/solvent-free (4:1)[18a]	**3b**	97 (30)

other noteworthy advantages of this method. Moreover, wide applicability and also excellent chemoselectivity make this reagent system a good choice for the use in the oxidation of sulfides.

EXPERIMENTAL

General

The sulfides **1a**, **1p**, **1q**, **1s**, **1t**, and **1d′** were purchased from Merck chemical company. The other sulfides were prepared according to the described procedure.[34] The yields refer to isolated pure products. Melting points were determined using a Mettler FP5 apparatus and are uncorrected. IR spectra were run on a Philips PU9716 spectrophotometer. ^{1}H NMR spectra were recorded on a Bruker 80 or 200 and or 500 MHz spectrometer in CDCl$_3$ as the solvent and TMS as internal standard. The elemental analysis was performed by Research Institute of Petroleum Industry, Tehran, Iran. BTPPDC was prepared according to the reported procedure.[32a]

Oxidation of Sulfides 1a–s to Sulfoxides 2a–s in Acetonitrile, General Procedure

To a solution of sulfide **1** (1 mmol) in CH$_3$CN (10 mL) in a round-bottomed flask (50 mL) equipped with a condenser and a magnetic stirrer, BTPPDC (1.28 g, 1.5 mmol) and AlCl$_3$ (0.134 g, 1 mmol) were added and refluxed for 40–240 min. The progress of the reaction was followed by TLC (eluent: *n*-hexane/ethyl acetate, 7:2). The mixture was filtered and the solid material was washed with CH_3CN (15 mL). The filtrate was evaporated and the resulting crude material was purifided by recrystallization or chromatography on silica-gel to afford the pure sulfoxide **2a–s** in 85–95% yields (Table III).

Oxidation of Sulfides 1a–s to Sulfoxides 2a–s Under Microwave Irradiation, General Procedure

A mixture of sulfide **1** (1 mmol), BTPPDC (1.028 g, 1.2 mmol) and $AlCl_3$ (0.134 g. 1 mmol) was prepared. CH_3CN (2 mL) was added and the mixture was exposed to microwave irradiation (900 W) for 0.75–5.5 min. The progress of the reaction was monitored by TLC (eluent: *n*-hexane/ethyl acetate, 7:2). After completion of the reaction, the mixture was extracted with ethyl acetate (2 × 20 mL). Evaporation of the solvent followed by recrystallization or chromatography on silica-gel afforded the pure sulfoxide **2a–s** in 85–96% yields (Table III).

Oxidation of Sulfides 1a–z, 1a′–d′ to Sulfones 3a–z, 3a′-d′ in Acetonitrile, General Procedure

In a round-bottomed flask equipped with a condenser and a magnetic stirrer, a solution of sulfide **1** (1 mmol) in CH_3CN (10 mL) was prepared. BTPPDC (2–3 mmol) and $AlCl_3$ (1 mmol) were added to the solution and the mixture was refluxed for 30–270 min. The progress of the reaction was followed by TLC (eluent: *n*-hexane/ethyl acetate, 7:1). The reaction mixture was filtered and the solid material was washed with CH_3CN (15 mL). The filtrate was evaporated and the crude product was either recrystallized or purified by chromatography on silica-gel to afford the pure sulfone **3a–z**, **3a′-d′** in 92–96% yields (Table IV).

Oxidation of Sulfides 1a–z, 1a′–d′ to Sulfones 3a–z, 3a′–d′ Under Microwave Irradiation, General Procdure

A mixture of sulfide **1** (1 mmol), BTPPDC (1.5–2.5 mmol), $AlCl_3$ (1 mmol) and CH_3CN (2 mL) was irradiated in a microwave oven (900 W) for 0.75–5.5 min. After completion of the reaction as indicated by TLC (eluent: *n*-hexane/ethyl acetate, 7:1), the reaction mixture was extracted with ethyl acetate (2 × 20 mL). The solvent was evaporated and the resulting crude material was purified by recrystallization or chromatography on silica-gel to afford the pure sulfone **3a–z**, **3a′–d′** in 93–98% yields (Table IV).

The physical and spectral data of the products are as follows.

Diphenyl Sulfoxide (2a)

M.p. 70–72°C (lit.[2] m.p. 69–72°C). IR (KBr): $\nu = 1038\ \mathrm{cm}^{-1}$. ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 7.41$–7.50 (m, 6H), 7.61–7.66 (m, 4H).

Benzyl Phenyl Sulfoxide (2b)

M.p. 122–123°C (lit.[2] m.p. 122–123°C). IR (KBr): $\nu = 1034\ \mathrm{cm}^{-1}$. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 4.04$ (d, 1H, J = 12.6 Hz), 4.14 (d, 1H, J = 12.6 Hz), 7.02–7.03 (m, 2H), 7.28–7.36 (m, 3H), 7.41–7.52 (m, 5H).

Dibenzyl Sulfoxide (2c)

M.p. 131–133°C (lit.[2] m.p. 132–134°C). IR (KBr): $\nu = 1026\ \mathrm{cm}^{-1}$. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 3.91$ (d, 2H, J = 13.0 Hz), 3.98 (d, 2H, J = 13.0 Hz), 7.31–7.41 (m, 10H).

4-Methylbenzyl Phenyl Sulfoxide (2d)

M.p. 97–99°C (lit.[35a] m.p. 99–100°C). IR (KBr): $\nu = 1027\ \mathrm{cm}^{-1}$. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 2.36$ (s, 3H), 3.99 (d, 1H, J = 12.6 Hz), 4.10 (d, 1H, J = 12.6 Hz), 6.91 (d, 2H, J = 7.70 Hz), 7.10 (d, 2H, J = 7.70 Hz), 7.42–7.51 (m, 5H).

4-Bromobenzyl Phenyl Sulfoxide (2e)

M.p. 178–179°C (lit.[35b] m.p. 179°C). IR (KBr): $\nu = 1029\ \mathrm{cm}^{-1}$. ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 3.98$ (d, 1H, J = 12.8 Hz), 4.05 (d, 1H, J = 12.8 Hz), 6.86 (d, 2H, J = 8.3 Hz), 7.31–7.51 (m, 7H).

4-Nitrobenzyl Phenyl Sulfoxide (2f)

M.p. 161–163°C. IR (KBr): $\nu = 1515, 1345, 1026\ \mathrm{cm}^{-1}$. ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 4.04$ (d, 1H, J = 12.0 Hz), 4.24 (d, 1H, J = 12.0 Hz), 7.13 (d, 2H, J = 6.5 Hz), 7.40–7.51 (m, 5H), 8.12 (d, 2H, J = 6.5 Hz). Anal. calcd. for $C_{13}H_{11}NSO_3$: C, 59.76; H, 4.24; N, 5.36; S, 12.27. Found: C, 59.63; H, 4.40; N, 5.25; S, 12.41.

Benzyl 4-Methylphenyl Sulfoxide (2g)

M.p. 121–123°C (lit.[30b] m.p. 122–124°C). IR (KBr): $\nu = 1024\ \mathrm{cm}^{-1}$. ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 2.40$ (s, 3H), 4.0 (d, 1H, J = 12.5 Hz), 4.10 (d, 1H, J = 12.5 Hz), 7.01–7.05 (m, 2H), 7.28–7.32 (m, 7H).

Benzyl 4-Bromophenyl Sulfoxide (2h)

M.p. 139–140°C (lit.[35b] m.p. 141–142°C). IR (KBr): $\nu = 1034\ \mathrm{cm}^{-1}$. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 4.0$ (d, 1H, J = 12.6 Hz), 4.1 (d, 1H, J = 12.6 Hz), 7.0 (d, 2H, J = 8.38 Hz), 7.24 (d, 2H, J = 8.38), 7.29–7.34 (m, 3H), 7.60 (d, 2H, J = 8.38 Hz).

Benzyl 4-Chlorophenyl Sulfoxide (2i)

M.p. 120–122°C (lit.[30b] m.p. 120–122°C). IR (KBr): $\nu = 1036$ cm$^{-1.}$ ^{1}H NMR (200 MHz, CDCl$_3$): $\delta = 4.02$ (d, 1H, J = 12.5 Hz), 4.15 (d, 1H, J = 12.5 Hz), 7.02 (d, 2H, J = 6.8 Hz), 7.26–7.46 (m, 5H), 7.36 (d, 2H, J = 6.8 Hz).

Benzyl 4-Bromobenzyl Sulfoxide (2j)

M.p. 139–140°C. IR (KBr): $\nu = 1028$ cm^{-1}. ^{1}H NMR (500 MHz, CDCl$_3$): $\delta = 3.78$ (d, 1H, J = 13.09 Hz), 3.89 (d, 1H, J = 13.09 Hz), 3.95 (s. 2H), 7.2 (d, 2H, J = 8.27 Hz), 7.32–7.33 (m, 2H), 7.40–7.44 (m, 3H), 7.54 (d, 2H, J = 8.27 Hz). Anal. calcd. for $C_{14}H_{13}SOBr$: C, 54.38; H, 4.24; S, 10.37. Found: C, 54.30; H, 4.22, S, 10.50.

4-Bromobenzyl 4-Methylphenyl Sulfoxide (2k)

M.p. 159–161°C (lit.[35] m.p. 161°C). IR (KBr): $\nu = 1030$ cm^{-1}. ^{1}H NMR (500 MHz, CDCl$_3$): $\delta = 2.46$ (s, 3H), 3.99 (s, 2H), 6.88 (d, 2H, J = 8.2 Hz), 7.27–7.31 (m, 4H), 7.42 (d, 2H, J = 8.2 Hz).

4-Bromobenzyl 4-Bromophenyl Sulfoxide (2l)

M.p. 143–145°C (lit.[35b] m.p. 145–146°C). IR (KBr): $\nu = 1038$ cm^{-1}. ^{1}H NMR (500 MHz, CDCl$_3$): $\delta = 3.98$ (d, 1H, J = 12.73 Hz), 4.02 (d, 1H, J = 2.73 Hz), 6.88 (d, 2H, J = 8.0 Hz), 7.26 (d, 2H, J = 8.0 Hz), 7.45 (d, 2H, J = 8.0 Hz), 7.62 (d, 2H, J = 8.0 Hz).

4-Bromobenzyl 4-Chlorophenyl Sulfoxide (2m)

M.p. 134–136°C (lit.[35b] m.p. 135–136°C). IR (KBr): $\nu = 1037$ cm^{-1}. ^{1}H NMR (200 MHz, CDCl$_3$): $\delta = 3.98$ (d, 1H, J = 12.85 Hz), 4.05 (d, 1H, J = 12.85 Hz), 6.87 (d, 2H, J = 8.40 Hz), 7.30 (d, 2H, J = 8.40 Hz), 7.45–7.51 (m, 4H).

4-Chlorophenyl 4-Nitrobenzyl Sulfoxide (2n)

M.p. 154–155°C (lit.[35c] m.p. 153.5–154.5°C). IR (KBr): $\nu = 1515, 1345, 1040$ cm^{-1}. ^{1}H NMR (500 MHz, CDCl$_3$): $\delta = 4.04$ (d, 1H, J = 12.83 Hz), 4.24 (d, 1H, J = 12.83 Hz), 7.17 (d, 2H, J = 8.53 Hz), 7.33 (d, 2H, J = 8.53 Hz), 7.48 (d, 2H, J = 8.53 Hz), 8.17 (d, 2H, J = 8.53 Hz).

2-Chlorobenzyl 4-Chlorophenyl Sulfoxide (2o)

M.p. 73–74°C (lit.[35b] m.p. 73–74°C). IR (KBr): $\nu = 1040$ cm^{-1}. ^{1}H NMR (500 MHz, CDCl$_3$): $\delta = 4.20$ (d, 1H, J = 12.56 Hz), 4.30 (d, 1H, J = 12.56 Hz), 7.16 (d, 2H, J = 7.05 Hz), 7.25 (t, 2H, J = 7.40 Hz), 7.41 (d, 2H, J = 8.5 Hz), 7.47 (d, 2H, J = 8.5 Hz).

Dipropyl Sulfoxide (2p)

Oil (lit.[34] oil). IR (neat): $\nu = 1020\ \text{cm}^{-1}$. ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 1.10$ (t, 6H, J = 7.2 Hz), 1.81–1.98 (m, 4H), 2.50–2.74 (m, 4H).

Dibutyl Sulfoxide (2q)

Oil (lit.[35i] oil). IR (neat): $\nu = 1022\ \text{cm}^{-1}$. ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 0.92$ (t, 6H, J = 7.2 Hz), 1.41–1.56 (m, 4H), 1.70–1.80 (m, 4H), 2.58–2.73 (m, 4H).

3-Methylbutyl n-Octyl Sulfoxide (2r)

M.p. 42–43°C. IR (KBr): $\nu = 1018\ \text{cm}^{-1}$. ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 0.88$–0.94 (m, 9H), 1.15–1.50 (m, 10H), 1.60–1.90 (m, 5H), 2.55–2.80 (m, 4H). Anal. calcd. for $C_{13}H_{28}SO$: C, 67.18; H, 12.14; S, 13.80. Found: C, 66.98; H, 12.08; S, 14.10.

Dibenzothiophene Sulfoxide (2s)

M.p. 181–183°C (lit.[2] m.p. 181–183°C). IR (KBr): $\nu = 1038\ \text{cm}^{-1}$. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 7.53$–7.70 (m, 4H), 7.83–8.04 (m, 4H).

Diphenyl Sulfone (3a)

M.p. 125–126°C (lit.[35i] m.p. 126–128°C). IR (KBr): $\nu = 1304$, 1150 cm^{-1}. ^{1}H NMR (80 MHz, $CDCl_3$): $\delta = 7.40$–7.60 (m, 6H), 7.85–8.04 (m, 4H).

Benzyl Phenyl Sulfone (3b)

M.p. 146–147°C (lit.[30a] m.p. 146–147°C). IR (KBr): $\nu = 1284$, 1120 cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 4.35$ (s, 2H), 7.10–7.14 (m, 2H), 7.30–7.37 (m, 3H), 7.44–7.69 (m, 5H).

Dibenzyl Sulfone (3c)

M.p. 148–149°C (lit.[34] m.p. 150°C). IR (KBr): $\nu = 1300$, 1122 cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 4.17$ (s, 4H), 7.39–7.44 (m, 10H).

4-Methylbenzyl Phenyl Sulfone (3d)

M.p. 150–152°C (lit.[35d] m.p. 151–152°C). IR (KBr): $\nu = 1298$, 1137 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 2.36$ (s, 3H), 4.31 (s, 2H), 7.0 (d, 2H, J = 7.9 Hz), 7.11 (d, 2H, J = 7.9 Hz), 7.50 (t, 2H, J = 7.8 Hz), 7.64 (t, 1H, J = 7.48 Hz), 7.68–7.69 (m, 2H).

4-Bromobenzyl Phenyl Sulfone (3e)

M.p. 195–197°C (lit.[35d] m.p. 191–193°C). IR (KBr): $\nu = 1300$, 1143 cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 4.37$ (s, 2H), 6.99 (d, 2H, J = 8.5 Hz), 7.42–7.80 (m, 7H).

4-Nitrobenzyl Phenyl Sulfone (3f)

M.p. 204–205°C. IR (KBr): $\nu = 1515$, 1345, 1300, 1142 cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 4.44$ (s, 2H), 7.10 (d, 2H, J = 6.5 Hz), 7.40–7.70 (m, 5H), 8.18 (d, 2H, J = 6.5 Hz). Anal. calcd. for $C_{13}H_{11}NSO_4$: C, 56.31; H, 3.99; N, 5.05; S, 11.56. Found: C, 56.17; H, 4.10; N, 4.96; S, 11.75.

Benzyl 4-Methylphenyl Sulfone (3g)

M.p. 143-144°C (lit.[30b] m.p. 142–144°C). IR (KBr): $\nu = 1308$, 1144 cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 2.40$ (s, 3H), 4.27 (s, 2H), 7.0-7.35 (m, 5H), 7.40–7.60 (m, 4H).

Benzyl 4-Bromophenyl Sulfone (3h)

M.p. 156–158°C (lit.[35b] m.p. 158–159°C). IR (KBr): $\nu = 1305$, 1140 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 4.35$ (s, 2H), 7.13 (d, 2H, J = 7.2 Hz), 7.31–7.38 (m, 3H), 7.50 (d, 2H, J = 8.5 Hz), 7.62 (d, 2H, J = 8.5 Hz).

Benzyl 4-Chlorophenyl Sulfone (3i)

M.p. 142–143°C (lit.[30b] m.p. 142–145°C). IR (KBr): $\nu = 1310$, 1143 cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$)$\delta = 4.35$ (s, 2H), 7.13 (d, 2H, J = 6.7 Hz), 7.31–7.6 (m, 5H), 7.72 (d, 2H, J = 6.7 Hz).

Benzyl 4-Bromobenzyl Sulfone (3j)

M.p. 176–178°C. IR (KBr): $\nu = 1299$, 1116 cm^{-1}. ^{1}H NMR (80 MHz, $CDCl_3$): $\delta = 4.02$ (s, 2H), 4.14 (s, 2H), 7.17–7.60 (m, 9H). Anal. calcd. for $C_{14}H_{13}BrSO_2$: C, 51.70; H, 4.03; S, 9.86. Found: C, 51.60; H, 4.10; S, 9.97.

4-Bromobenzyl 4-Methylphenyl Sulfone (3k)

M.p. 172–173°C (lit.[35c] m.p. 171–172°C). IR (KBr): $\nu = 1299$, 1140 cm^{-1}. ^{1}H NMR (80 MHz, $CDCl_3$): $\delta = 2.41$ (s, 3H), 4.24 (s, 2H), 7.0 (d, 2H, J = 8.2 Hz), 7.27–7.31 (m, 4H), 7.42 (d, 2H, J = 8.2 Hz).

4-Bromobenzyl 4-Bromophenyl Sulfone (3l)

M.p. 179–180°C (lit.[35b] m.p. 179–180°C). IR (KBr): $\nu = 1304$, 1144 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 4.29$ (s, 2H), 7.02 (d, 2H, J = 8.45 Hz), 7.47 (d, 2H, J = 8.45 Hz), 7.53 (d, 2H, J = 8.45 Hz), 7.67 (d, 2H, J = 8.45 Hz).

4-Bromobenzyl 4-Chlorophenyl Sulfone (3m)

M.p. 157–158°C (lit.[35b] m.p. 158–159°C). IR (KBr): $\nu = 1303$, 1144 cm^{-1}. ^{1}H NMR (200 MHz, $CDCl_3$): $\delta = 4.29$ (s, 2H), 7.01 (d, 2H, J = 8.26 Hz), 7.25–7.63 (m, 6H).

4-Chlorophenyl 4-Nitrobenzyl Sulfone (3n)

M.p. 174–175°C (lit.[35c] m.p. 175–176°C). IR (KBr): $\nu = 1515$, 1345, 1300, 1140 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 4.44$ (s, 2H), 7.37 (d, 2H, J = 8.6 Hz), 7.52 (d, 2H, J = 8.6 Hz), 7.64 (d, 2H, J = 8.6 Hz), 8.21 (d, 2H, J = 8.6 Hz).

2-Chlorobenzyl 4-Chlorophenyl Sulfone (3o)

M.p. 120–121°C (lit.[35b] m.p. 120–121°C). IR (KBr): $\nu = 1310$, 1130 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 4.60$ (s, 2H), 7.28–7.36 (m, 3H), 7.45 (d, 2H, J = 7.76 Hz), 7.49–7.54 (m, 1H), 7.59 (d, 2 H, J = 7.76 Hz).

Dipropyl Sulfone (3p)

Oil (lit.[34] oil). IR (neat): $\nu = 1269$, 1130 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 1.06$ (t, 6H, J = 7.5 Hz), 1.79–1.85 (m, 4H), 2.88 (t, 4H, J = 7.96 Hz).

Dibutyl Sulfone (3q)

M.p. 44–45°C (lit.[35i] m.p. 43–45°C). IR (KBr): $\nu = 1259$, 1120 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 1.01$ (t, 6H, J = 7.36 Hz), 1.48–1.57 (m, 4H), 1.82–1.88 (m, 4H), 2.97 (t, 4H, J = 8.10 Hz).

3-Methylbutyl n-Octyl Sulfone (3r)

M.p. 39–41°C. IR (KBr): $\nu = 1266$, 1110 cm^{-1}. ^{1}H NMR (80 MHz, $CDCl_3$): $\delta = 0.80$–1.90 (m, 24 H), 2.65–2.90 (m, 4 H). Anal. calcd. for $C_{13}H_{28}SO_2$: C, 62.85; H, 11.36; S, 12.91. Found: C, 62.70; H, 11.45; S, 13.10.

Dibenzothiophene Sulfone (3s)

M.p. 232–234°C (lit.[35i] m.p. 231–233°C). IR (KBr): $\nu = 1282$, 1160 cm^{-1}. ^{1}H NMR (80 MHz, $CDCl_3$): $\delta = 7.04$–7.60 (m, 4H), 7.62–7.90 (m, 4H).

Thioxanthen-9-One 10,10-Dioxide (3t)

M.p. 186–187°C (lit.[35g] m.p. 187–188°C). IR (KBr): $\nu = 1677$, 1290, 1158 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 7.84$–7.93 (m, 4H), 8.22–8.40 (m, 4H).

Methyl n-Ocyl Sulfone (3u)

M.p. 63–64°C. IR (KBr): $\nu = 1275$, 1122 cm^{-1}. ^{1}H NMR (80 MHz, $CDCl_3$): $\delta = 0.80$–1.85 (m, 15H), 2.70–3.0 (m, 5H). Anal. calcd. for $C_9H_{20}SO_2$: C, 56.21; H, 10.48; S, 16.67. Found: C, 56.15; H, 10.40; S, 16.85.

Ethyl n-Octyl Sulfone (3v)

M.p. 66–67°C. IR (KBr): $\nu = 1272$, 1114 cm^{-1}. ^{1}H NMR (80 MHz, $CDCl_3$): $\delta = 0.80$–1.85 (m, 18H), 2.65–3.0 (m, 4H). Anal. calcd. for $C_{10}H_{22}SO_2$: C, 58.21; H, 10.74; S, 15.54. Found: C, 58.20; H, 10.70; S, 15.65.

Methyl Phenyl Sulfone (3w)

M.p. 84–85°C (lit.[30b] m.p. 85–86°C). IR (KBr): $\nu = 1295$, 1140 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 3.10$ (s, 3H), 7.61–7.72 (m, 3H), 7.99–8.0 (m, 2H).

Methyl 4-Methylphenyl Sulfone (3x)

M.p. 84–86°C (lit.[35h] m.p. 85–87°C). IR (KBr): $\nu = 1314$, 1139 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 2.49$ (s, 3H), 3.07 (s, 3H), 7.40 (d, 2H, J = 8.0 Hz), 7.86 (d, 2H, J = 8.0 Hz).

4-Chlorophenyl Methyl Sulfone (3y)

M.p. 95–97°C. IR (KBr): $\nu = 1304$, 1142 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 3.10$ (s, 3H), 7.60 (d, 2H, J = 8.5 Hz), 7.90 (d, 2H, J = 8.5 Hz). Anal. calcd. for $C_7H_7ClSO_2$: C, 44.10; H, 3.70; S, 16.82. Found: C, 43.90; H, 3.80; S, 17.10.

Phenyl n-Propyl Sulfone (3z)

M.p. 44–45°C (lit.[35e] m.p. 46°C). IR (KBr): $\nu = 1304$, 1140 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 1.06$ (t, 3H, J = 6 Hz), 1.71–1.84 (m, 2H), 3.10 (t, 2H, J = 6 Hz), 7.53–7.60 (m, 3H), 7.93–7.94 (m, 2H).

n-Butyl Phenyl Sulfone (3a′)

Oil (lit.[35e] oil). IR (neat): $\nu = 1300$, 1140 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 0.96$ (t, 3H, J = 6 Hz), 1.44–1.73 (m, 4H), 3.10 (t, 2H, J = 6 Hz), 7.53–7.60 (m, 3H), 7.93–7.94 (m, 2H).

Phenyl 2-Propyl Sulfone (3b′)

Oil (lit.[35e] oil). IR (neat): $\nu = 1300, 1140\ \mathrm{cm}^{-1}$. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 1.31$ (d, 6H, J = 6.3 Hz), 3.14 (q, 1H, J = 6.2 Hz), 7.49–7.60 (m, 3H), 7.88–7.89 (m, 2H).

Allyl Phenyl Sulfone (3c′)

Oil (lit.[30b] oil). IR (neat): $\nu = 1667, 1307, 1140\ \mathrm{cm}^{-1}$. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 3.80$ (d, 2H, J = 7 Hz), 5.16 (d, 1H, J = 17 Hz), 5.36 (d, 1H, J = 10.2 Hz), 5.74–5.86 (m, 1H), 7.40–7.65 (m, 5H).

Diallyl Sulfone (3d′)

Oil (lit.[35f] oil). IR (neat): $\nu = 1639, 1314, 1124\ \mathrm{cm}^{-1}$. ^{1}H NMR (500 MHz, $CDCl_3$): $\delta = 3.70$ (d, 4H, J = 7.30 Hz), 5.40–5.54 (m, 4H), 5.90–6.0 (m, 2H).

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