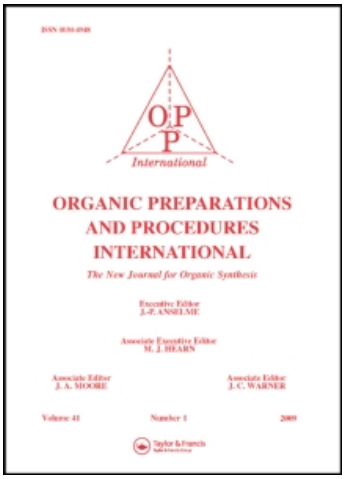

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Potassium Fluoride as an Efficient and Reusable Reagent for the Synthesis of *N,N*-Dialkylsulfonamides *via Aza*-Conjugate Addition Reaction Under Microwave Irradiation

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Potassium Fluoride as an Efficient and Reusable Reagent for the Synthesis of *N*,*N*-Dialkylsulfonamides *via Aza*-Conjugate Addition Reaction Under Microwave Irradiation

Alireza Hasaninejad,[1] Abdolkarim Zare,[2] Abolfath Parhami,[2] Ahmad Reza Moosavi-Zare,[2] Raheleh Bargebid,[3] Mohammad Hassan Beyzavi,[3] Ali Khalafi-Nezhad,[3] Azam Arghoon,[2] Maria Merajoddin,[2] Sayyed Ali Moosavi,[2] Abdoreza Dara,[2] and Mohsen Shekouhy[1]

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N,*N*-Dialkyl derivatives of sulfonamides are significant due to their potential applications as anti-ulcer, antidepressant, anti-emetic analgesic, psychostimulant and anti-inflammatory agents.[1] *Aza*-conjugate addition of sulfonamides to electrophilic double bonds can be a useful synthetic route toward *N*,*N*-dialkylsulfonamides. Indeed, to the best of our knowledge, this transformation has scarcely been studied in the literature. Reagents such as Al_2O_3,[2] ZnO,[3] MgO,[4] K_2CO_3[5] and PBu_3[6] have been applied to achieve *aza*-conjugate addition reaction of sulfonamides. However, most of these reagents are useful for the synthesis of *N*-alkylsulfonamides but not of *N*,*N*-dialkylated adducts.[2–5] Therefore, it seems highly desirable to develop an efficient reagent for the preparation of *N*,*N*-dialkylsulfonamides. Solvent-free organic reactions have become as a useful protocol in organic synthesis.[7,8] The utilization of microwave irradiation coupled with solvent-free reaction conditions, provides chemical processes with special attributes, such as enhanced reaction rates, higher yields, easier work-up, better selectivity, and improved ease of manipulation.[7,8] KF immobilized onto supports possessing high-surface area or accompanied with other reagents has been extensively used in organic synthesis;[9–19] however, there are only a few reports on the applications of KF itself as reagent in the literature.[20,21] In extension of our previous studies

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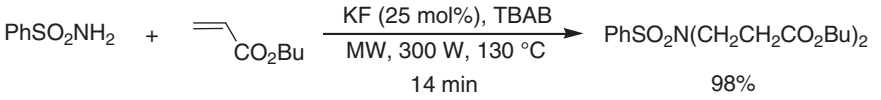
on the synthesis of sulfonamide derivatives[3–5] as well as green organic synthesis,[3–5,22–27] herein we report an efficient method for the preparation of *N*,*N*-dialkylsulfonamides *via aza*-conjugate addition of sulfonamides to α,β-unsaturated esters in the presence of KF and tetrabutylammonium bromide (TBAB) under microwave irradiation in the absence of solvent (*Scheme 1*).



Scheme 1

The *aza*-conjugate addition of benzenesulfonamide (2 mmol) to *n*-butyl acrylate (4.4 mmol) in the presence of different molar ratios of KF and TBAB (1 mmol) at range of 100–500 W of microwave power (max. 130°C) (*Scheme 1*) was studied in order to determine the optimal conditions with respect to molar ratio of the reagent to the substrate and microwave power. The data summarized in *Table 1* indicates that reasonable results were obtained when the reaction was carried out using 25 mol% KF at 300 W of microwave power. Under these conditions, only *N*,*N*-dialkylsulfonamide was produced in 98% yield, and no mono-*N*-alkylated product was formed. The reaction was also examined in the absence of KF; however, these conditions were not efficient (*Table 1*). In fact, this reaction requires a base to abstract a proton from the amino group of the sulfonamide thus generating the nucleophile to attack the β-carbon of the α,β-unsaturated compound.[16,21,28–30] In this study, it was found that potassium fluoride can efficiently serve as the base.[16,21,28–30]

To assess the efficiency of microwave heating in comparison with conventional heating, the reaction of benzenesulfonamide (2 mmol) with *n*-butyl acrylate (4.4 mmol) in the presence of KF (25 mol%) and TBAB (1 mmol) was examined in an oil-bath (130°C). Under these conditions, the selectivity decreased and the *N*,*N*-dialkyl and *N*-alkyl sulfonamides were obtained in 87 and 6% respectively after 3 hrs. Thus, microwave irradiation has an

Table 1
Influence of Ratios of KF to the Substrate and Microwave Power in the Presence of TBAB

			Yield (%)	
Molar Ratio of KF (mol%)	MW Power (W)	Time (min)	N,N-Dialkylated Product	N-Alkylated Product
0	300	30	7	29
15	300	20	84	10
25	300	14	98	—
35	300	11	98	—
25	100	25	47	23
25	200	20	73	15
25	400	9	96	—
25	500	5	90	—

Table 2
Comparative Reaction between Benzenesulfonamide and *n*-Butyl Acrylate using KF in Various Solvents versus the Solvent-Free Method under Microwave Irradiation

			Yield (%)	
Entry	Solvent	Time (min)	*N*,*N*-Dialkyl Product	*N*-Alkyl Product
1	DMSO	18	82	9
2	DMF	18	94	4
3	HMPTA	20	79	7
4[a]	Solvent-free	14	98	—
5[b]	Solvent-free	25	19	12
6[c]	Solvent-free	14	73	Trace

[a]Solvent-free conditions in the presence of TBAB.
[b]Solvent-free conditions without TBAB.
[c]Solvent-free conditions using TBAF.

essential influence on the progress and the selectivity of the aza-conjugate addition reaction. In another study, the effectiveness of the solvent-free method versus solution conditions was investigated (*Table 2*). For this purpose, a mixture of benzenesulfonamide (2 mmol), *n*-butyl acrylate (4.4 mmol), KF (25 mol%) and TBAB (1 mmol) was irradiated in a microwave oven (300 W, max. 130°C) in various solvents (4 mL). Higher yield of the *N*,*N*-dialkylated product in shorter reaction times were obtained in the solvent-free conditions. The reaction was also examined in the absence of TBAB under solventless conditions; however, these conditions were not efficient (see *Table 2*, *Entry 5*). Thus, the presence of TBAB in the reaction is necessary. In general, TBAB melts at 100°C and solubilizes the reactants.[5,25,31] Moreover, this quaternary ammonium salt absorbs the microwave irradiation and increases the reaction temperature.[5,25] Moreover, the reaction of benzenesulfonamide with *n*-butyl acrylate was checked using tetrabutylammonium fluoride (TBAF) without KF under solvent-free conditions at 300 W (max. 130°C). Under these conditions, the product was obtained in 73% after 14 min (*Table 2*, *Entry 6*).

To evaluate the efficiency and potency of our method with respect to the reported methods for the synthesis of *N*,*N*-dialkylsulfonamides *via aza*-conjugate addition reaction, we have tabulated the results of the reported methods for the synthesis of *N*,*N*-di(3-carbo-*n*-butoxyethyl)benzenesulfonamide in *Table 3*. Clearly, in comparison with the reported methods, our method significantly improved the synthesis of *N*,*N*-dialkylated sulfonamides *via aza*-conjugate addition reaction.

To determine the generality and scope of the method, the reaction was examined with different sulfonamides and α,β-unsaturated esters (*Table 4*). The reactions proceeded efficiently and the desired conjugate adducts were obtained in good to excellent yields in short reaction times. The nature of the alkoxy group of α,β-unsaturated esters had a negligible effect on the reaction results (*Entries 1–8*). With sterically hindered ethyl methacrylate, lower yields were obtained even under prolonged reaction times (*Entries 9* and *10*) while with ethyl crotonate, only the *N*-monoalkyl product was isolated in 92%

Table 3

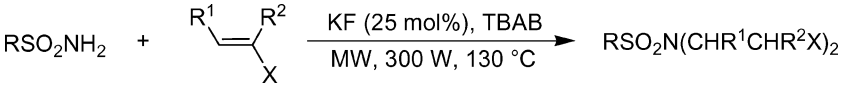
Synthesis of *N*,*N*-di(3-carbo-*n*-butoxyethyl)benzenesulfonamide using the reported methods compared to Present Method

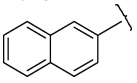
Entry	Reagent	Time (min)	Yield (%)	Ref.
1	KF	14	98	Present Work
2	ZnO	5	9	3
3	MgO	5	7	4
4	K_2CO_3	5	16	5
5[a]	PBu_3	1080	52	6

[a]Yield of *N*,*N*-dialkylsulfonamide from *p*-toluenesulfonamide and ethyl acrylate.

Table 4

Synthesis of *N*,*N*-Dialkylsulfonamides under Solvent-free and Microwave Irradiation



Entry	R	R^1	R^2	X	Time (min)	Yield (%)	m.p. (lit.)
1[a]	C_6H_5	H	H	$CO_2CH_2CH_3$	27	96	Oil (Oil)[3]
2	C_6H_5	H	H	$CO_2CH_2(CH_2)_2CH_3$	14	98	Oil (Oil)[3]
3	C_6H_5	H	H	$CO_2CH_2(CH_2)_4CH_3$	14	97	Oil (Oil)[3]
4	C_6H_5	H	H	$CO_2CH_2C_6H_5$	15	97	Oil (Oil)[4]
5	C_6H_5	H	H	$CO_2CH_2CH_2C_6H_5$	17	97	Oil (Oil)[4]
6	C_6H_5	H	H	$CO_2CH_2CH=CHC_6H_5$	18	95	Oil (Oil)[4]
7	*p*-$CH_3C_6H_4$	H	H	$CO_2CH_2(CH_2)_2CH_3$	17	97	Oil (Oil)[5]
8	*p*-$CH_3C_6H_4$	H	H	$CO_2CH_2C_6H_5$	18	95	Oil
9[b]	C_6H_5	H	CH_3	$CO_2CH_2CH_3$	20	79	Oil (Oil)[5]
10[b]	*p*-$CH_3C_6H_4$	H	CH_3	$CO_2CH_2CH_3$	20	77	Oil
11[c]	C_6H_5	CH_3	H	$CO_2CH_2CH_3$	30	—	—
12		H	H	$CO_2CH_2(CH_2)_2CH_3$	14	97	Oil
13	CH_3	H	H	$CO_2CH_2CH_2C_6H_5$	20	64	Oil
14[d]	*p*-$CH_3C_6H_4$	H	H	CN	30	39	97–99 (102–104)[6]
15[d]	C_6H_5	H	H	$COCH_3$	30	Trace	—
16[d]	C_6H_5	H	H	CHO	30	—	—
17[e]	C_6H_5	H	H	$CONH_2$	30	—	—

[a]Because of lower boiling point of ethyl acrylate, this reaction was performed at 100°C (300 W of microwave power); moreover, in this case, ethyl acrylate/benzenesulfonamide ratio (mol/mol) was 2.5/1.

[b]This reaction was carried out at 400 W (max. 140°C), and in this case, 100 mol% of KF and TBAB were used.

[c]In this reaction, only mono-*N*-alkyl product was produced in 92%.

[d]This reaction was carried out in a capped vessel at 100°C, and α,β-unsaturated compound/benzenesulfonamide ratio (mol/mol) was 3/1.

[e]This reaction was examined at 120°C.

yield after 30 min (*Entry 11*). *p*-Toluenesulfonamide and 2-naphthylsulfonamide afforded the expected products in excellent yields (*Entries 7*, *8*, *10* and *12*). Moreover, the method was successful for *N*,*N*-dialkylation of alkysulfonamides (*Entry 13*). The method was also examined for the reaction of sulfonamides with α,β-unsaturated nitriles, ketones, aldehydes and amides. As *Table* 4 indicates, while acrylonitrile gave the corresponding *N*,*N*-dialkylsulfonamide in moderate yield (*Entry 14*), the reaction was not successful with other α,β-unsaturated compounds (*Entries 15–17*). One of the most interesting properties of KF is its ease of recycling. The yields of *N*,*N*-di(3-carbo-*n*-butoxyethyl)benzenesulfonamide (model compound) in the 2nd and 3rd and 4th uses of the reagent were nearly as high as in the first use.

In summary, we have developed a new method for the synthesis of *N*,*N*-dialkylsulfonamides via *aza*-conjugate addition of sulfonamides to α,β-unsaturated esters promoted by microwave irradiation. The advantages of this method are generality, high yield, high selectivity, short reaction time, low cost, ease of product isolation, and agreement with the green chemistry protocols.

Experimental Section

All chemicals were purchased from Merck or Fluka Chemical Companies. All known compounds were identified by comparison of their spectral data with those in the authentic samples. All reactions were carried out using laboratory microwave oven (MW 3000, Landgraf Company, Germany). IR spectra were run on a Shimadzu FTIR-8300 spectrophotometer. The ^{1}H NMR (250 MHz) and ^{13}C NMR (62.5 mhz) were run on a Bruker Avance DPX-250, FT-NMR spectrometer. Microanalysis was performed on a Perkin-Elmer 240-B microanalyzer. Mass spectra were recorded on a Shimadzu GC MS-QP 1000 EX apparatus.

General Procedure for the Synthesis of N,N-Dialkylsulfonamides

To a well-ground mixture of the sulfonamide (2 mmol), KF (0.029 g, 0.5 mmol) and TBAB (0.322 g, 1 mmol) in a microwave vessel was added the α,β-unsaturated ester (4.4 mmol) and mixed thoroughly with a glass rod (the microwave vessel was open). The resulting mixture was irradiated in a microwave oven at 300 W at a maximum internal temperature of 130°C (the internal reaction temperature was measured by a sensor that was inserted in the reaction mixture). After each 4 min, the microwave irradiation was stopped and the reaction mixture was rapidly mixed with a glass rod. After completion of the reaction monitored with TLC, the reaction mixture was cooled to room temperature and suspended in EtOAc (40 mL), filtered to separate the reagent and the filtrate was washed with water (2×60 mL) and dried with $MgSO_4$. The solvent was evaporated and the crude product was purified by short column chromatography on silica gel eluted with EtOAc/*n*-hexane (1/3). After isolation of the products, the remaining KF was washed with EtOAc, dried and used for the next run under identical reaction conditions.

***N*,*N*-Di(3-carbethoxyethyl)benzenesulfonamide (*Table 4*, *Entry 1*):** Colorless oil, (lit.[3] oil); IR (neat): 3028, 2984, 1733, 1447, 1317 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 1.22 (t, $J = 7.1$ Hz, 6H, $2CH_3$), 2.59 (t, $J = 5.0$ Hz, 4H, $2O{=}CCH_2$), 3.45 (t, $J = 5.0$ Hz, 4H, $2O{=}CCH_2CH_2$), 4.08 (q, $J = 7.1$ Hz, 4H, $2OCH_2$), 7.46–7.57 (complex, 3H, H_3-H_5 of the

aromatic ring), 7.87 (m, 2H, H_2 and H_6 of the aromatic ring); ^{13}C NMR ($CDCl_3$): δ 13.9, 32.8, 44.7, 60.4, 127.2, 129.1, 132.6, 138.9, 170.9; MS (m/z): 357 (M^+).

***N,N*-Di(3-carbo-*n*-butoxyethyl)benzenesulfonamide (*Table 4*, *Entry 2*):** Colorless oil, (lit.[3] oil); IR (neat): 3036, 2966, 1732, 1447, 1317 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 0.92 (t, $J = 6.5$ Hz, 6H, $2CH_3$), 1.33 (m, 4H, $2CH_3CH_2$),1.56 (m, 4H, $2CH_3CH_2CH_2$), 2.63 (t, $J = 5.1$ Hz, 4H, $2O{=}CCH_2$), 3.45 (t, $J = 5.1$ Hz, 4H, $2O{=}CCH_2CH_2$), 4.06 (t, $J = 6.9$ Hz, 4H, $2OCH_2$), 7.51–7.60 (complex, 3H, H_3-H_5 of the aromatic ring), 7.82 (m, 2H, H_2 and H_6 of the aromatic ring); ^{13}C NMR ($CDCl_3$): δ 13.5, 18.9, 30.5, 34.2, 44.4, 64.2, 126.9, 129.0, 132.4, 138.9, 171.1; MS (m/z): 413 (M^+).

***N,N*-Di(3-carbo-*n*-hexyloxyethyl)benzenesulfonamide (*Table 4*, *Entry 3*):** Colorless oil, (lit.[3] oil); IR (neat): 3031, 2974, 1733, 1447, 1316 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 0.89 (t, $J = 6.6$ Hz, 6H, $2CH_3$), 1.26–1.32 (complex, 12H, $2CH_3CH_2$, $2CH_3CH_2CH_2$ and $2CH_3(CH_2)_2CH_2$), 1.58 (m, 4H, $2CH_3(CH_2)_3CH_2$), 2.61 (t, $J = 5.0$ Hz, 4H, $2O{=}CCH_2$), 3.41 (t, $J = 5.0$ Hz, 4H, $2O{=}CCH_2CH_2$), 4.05 (t, $J = 6.9$ Hz, 4H, $2OCH_2$), 7.43–7.50 (complex, 3H, H_3-H_5 of the aromatic ring), 7.74 (m, 2H, H_2 and H_6 of the aromatic ring); ^{13}C NMR ($CDCl_3$): δ 13.8, 22.3, 25.5, 28.0, 30.4, 33.9, 44.5, 64.4, 126.4, 128.9, 132.1, 139.4, 171.4; MS (m/z): 392 (M^+-C_6H_5), 368 (M^+-$C_6H_{13}O$), 328 (M^+-$C_6H_5SO_2$).

***N,N*-Di(3-carbobenzyloxyethyl)benzenesulfonamide (*Table 4*, *Entry 4*):** Pale yellow oil, (lit.[4] oil); IR (neat): 3051, 2965, 1734, 1447, 1316 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 2.58 (t, $J = 5.0$ Hz, 4H, $2O{=}CCH_2$), 3.36 (t, $J = 5.0$ Hz, 4H, $2O{=}CCH_2CH_2$), 5.00 (s, 4H, $2OCH_2$), 7.22–7.25 (complex, 10H, $2C_6H_5CH_2$), 7.39–7.48 (complex, 3H, H_3-H_5 of the aromatic ring of sulfonamide moiety), 7.72 (m, 2H, H_2 and H_6 of the aromatic ring of sulfonamide moiety); ^{13}C NMR ($CDCl_3$): δ 33.3, 43.9, 65.5, 126.2, 127.7, 128.1, 128.9, 129.5, 131.8, 134.4, 137.8, 171.4; MS (m/z): 404 (M^+-C_6H_5), 340 (M^+-$C_6H_5SO_2$).

***N,N*-Di(3-carbophenethyloxyethyl)benzenesulfonamide (*Table 4*, *Entry 5*):** Pale yellow oil, (lit.[4] oil); IR (neat): 3045, 2942, 1733, 1447, 1331 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 2.51 (t, $J = 5.2$ Hz, 4H, $2O{=}CCH_2$), 2.82 (t, $J = 6.9$ Hz, 4H, $2ArCH_2$), 3.30 (t, $J = 5.2$ Hz, 4H, $2O{=}CCH_2CH_2$), 4.19 (t, $J = 6.9$ Hz, 4H, $2OCH_2$), 7.10–7.19 (complex, 10H, $2C_6H_5CH_2$), 7.42–7.45 (complex, 3H, H_3-H_5 of the aromatic ring of sulfonamide moiety), 7.78 (m, 2H, H_2 and H_6 of the aromatic ring of sulfonamide moiety); ^{13}C NMR ($CDCl_3$): δ 34.3, 34.9, 44.9, 65.2, 126.6, 126.7, 126.9, 128.6, 128.8, 129.2, 132.8, 137.5, 171.0; MS (m/z): 432 (M^+-C_6H_5), 388 (M^+-C_8H_9O), 368 (M^+-$C_6H_5SO_2$).

***N,N*-Di(3-carbocinnamyloxyethyl)benzenesulfonamide (*Table 4*, *Entry 6*):** Pale yellow oil, (lit.[4] oil); IR (neat): 3026, 2981, 1732, 1448, 1317 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 2.51 (t, $J = 5.1$ Hz, 4H, $2O{=}CCH_2$), 3.41 (t, $J = 5.1$ Hz, 4H, $2O{=}CCH_2CH_2$), 4.73 (m, 4H, $2OCH_2$), 6.15 (m, 2H, $2PhCH = CH$), 6.54 (d, 2H $J = 15.7$ Hz, PhC*H*), 7.28–7.36 (complex, 10H, $2C_6H_5CH_2$), 7.41–7.47 (complex, 3H, H_3-H_5 of the aromatic ring of sulfonamide moiety), 7.94 (m, 2H, H_2 and H_6 of the aromatic ring of sulfonamide moiety); ^{13}C NMR ($CDCl_3$): δ 33.8, 43.2, 65.4, 122.4, 123.0, 126.7, 126.9, 127.9, 128.5, 129.4, 132.4, 134.1, 139.3, 171.5; MS (m/z): 392 (M^+-$C_6H_5SO_2$).

***N,N*-Di(3-carbo-*n*-butoxyethyl)-*p*-toluenesulfonamide (*Table 4*, *Entry 7*):** Pale yellow oil, (lit.[5] oil); IR (neat): 3056, 2975, 1734, 1317 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 0.92 (t, $J = 6.9$ Hz, 6H, $2CH_2CH_3$), 1.32 (m, 4H, $2CH_3CH_2$),1.57 (m, 4H, $2CH_3CH_2CH_2$), 2.42 (s, 3H, $ArCH_3$), 2.63 (t, $J = 5.2$ Hz, 4H, $2O{=}CCH_2$), 3.43 (t, $J = 5.2$ Hz, 4H, $2O{=}CCH_2CH_2$), 4.02 (t, $J = 7.0$ Hz, 4H, $2OCH_2$), 7.31 (d, $J = 7.9$ Hz, 2H, H_3 and H_5 of the aromatic

ring), 7.72 (d, $J = 7.9$ Hz, 2H, H_2 and H_6 of the aromatic ring); ^{13}C NMR ($CDCl_3$): δ 13.6, 19.0, 21.4, 30.4, 33.9, 45.0, 64.5, 126.9, 129.5, 135.9, 143.5, 172.5; MS (m/z): 427 (M^+).

***N,N*-Di(3-carbbenzyloxyethyl)-*p*-toluenesulfonamide (*Table 4*, *Entry 8*):** Pale yellow oil; IR (neat): 3056, 2971, 1734, 1316 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 2.38 (s, 3H, $ArCH_3$), 2.60 (t, $J = 5.2$ Hz, 4H, 2O=CCH_2), 3.39 (t, $J = 5.2$ Hz, 4H, 2O=CCH_2CH_2), 5.04 (s, 4H, $2OCH_2$), 7.18–7.22 (complex, 10H, $2C_6H_5CH_2$), 7.34 (d, $J = 7.9$ Hz, 2H, H_3 and H_5 of the aromatic ring of sulfonamide moiety), 7.74 (d, $J = 7.9$ Hz, 2H, H_2 and H_6 of the aromatic ring of sulfonamide moiety); ^{13}C NMR ($CDCl_3$): δ 22.2, 33.7, 44.5, 65.8, 126.3, 127.1, 128.3, 129.0, 129.9, 134.9, 135.6, 143.1, 171.9; MS (m/z): 340 (M^+-$C_7H_7SO_2$).

Anal. for $C_{27}H_{29}NO_6S$: C, 65.44; H, 5.90; N, 2.83. Found: C, 65.63; H, 6.04; N, 2.71.

***N,N*-Di(3-carbethoxy-2-methyethyl)benzenesulfonamide (*Table 4*, *Entry 9*):** Pale yellow oil, (lit.[5] oil); IR (neat): 3042, 2982, 1732, 1447, 1316 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 1.09–1.19 (complex, 12H, $4CH_3$), 2.78 (m, 2H, 2O=CCH), 3.22–3.27 (complex, 4H, 2O=CCH_2CH_2), 4.03 (q, $J = 7.0$ Hz, 4H, $2OCH_2$), 7.44–7.53 (complex, 3H, H_3-H_5 of the aromatic ring), 7.75 (m, 2H, H_2 and H_6 of the aromatic ring); ^{13}C NMR ($CDCl_3$): δ 14.1, 15.3, 39.2, 52.4, 60.7, 127.3, 129.1, 132.7, 139.0, 174.7; MS (m/z): 385 (M^+).

***N,N*-Di(3-carbethoxy-2-methyethyl)-*p*-toluenesulfonamide (*Table 4*, *Entry 10*):** Pale yellow oil; IR (neat): 3057, 2973, 1733, 1447, 1316 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 1.12–1.24 (complex, 12H, $4CH_3$), 2.44 (s, 3H, $ArCH_3$), 2.73 (m, 2H, 2O=CCH), 3.25–3.31 (complex, 4H, 2O=CCH_2CH_2), 4.10 (q, $J = 7.0$ Hz, 4H, $2OCH_2$), 7.33 (d, $J = 7.8$ Hz, 2H, H_3 and H_5 of the aromatic ring), 7.76 (d, $J = 7.8$ Hz, 2H, H_2 and H_6 of the aromatic ring); ^{13}C NMR ($CDCl_3$): δ 13.9, 15.0, 22.7, 39.4, 51.2, 61.2, 126.6, 129.7, 136.1, 142.95, 172.1; MS (m/z): 399 (M^+).

Anal. for $C_{19}H_{29}NO_6S$: C, 57.12; H, 7.32; N, 3.51. Found: C, 56.87; H, 7.21; N, 3.65.

***N,N*-Di(3-carbo-*n*-butoxyethyl)-2-naphthylsulfonamide (*Table 4*, *Entry 12*):** Pale yellow oil; IR (neat): 3046, 2981, 1732, 1448, 1327 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 0.90 (t, $J = 6.6$ Hz, 6H, $2CH_3$), 1.36 (m, 4H, $2CH_3CH_2$), 1.57 (m, 4H, $2CH_3CH_2CH_2$), 2.52 (t, 4H, $J = 5.1$ Hz, 2O=CCH_2), 3.43 (t, 4H, $J = 5.1$ Hz, 2O=CCH_2CH_2), 4.06 (t, 4H, $J = 7.0$ Hz, $2OCH_2$), 7.49–7.55 (complex, 4H), 7.76–7.78 (complex, 3H); ^{13}C NMR ($CDCl_3$): δ 13.7, 19.1, 30.2, 33.6, 44.8, 64.8, 122.1, 128.0, 127.7, 128.0, 128.2, 128.5, 128.9, 129.1, 129.6, 132.3, 171.8; MS (m/z): 390 (M^+-C_4H_9O), 272 (M^+-$C_{10}H_7SO_2$).

Anal. for $C_{24}H_{33}NO_6S$: C, 62.18; H, 7.17; N, 3.02. Found: C, 62.01; H, 7.05; N, 3.18.

***N,N*-Di(3-carbophenethyloxyethyl)methanesulfonamide (*Table 4*, *Entry 13*):** Pale yellow oil; IR (neat): 3061, 2973, 1732, 1446, 1329, 1161 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 1.67 (s, 3H, CH_3), 2.80–2.89 (m, 8H), 3.81–3.88 (m, 8H), 7.21-.7.32 (complex, 10H, $2C_6H_5$); ^{13}C NMR ($CDCl_3$): δ 35.0, 35.2, 39.2, 63.7, 64.9, 126.6, 128.6, 129.0, 138.5, 170.6; MS (m/z): 368 (M^+-CH_3SO_2).

Anal. for $C_{23}H_{29}NO_6S$: C, 61.72; H, 6.53; N, 3.13. Found: C, 62.01; H, 6.72; N, 2.97.

***N,N*-Di(2-cyanoethyl)-*p*-toluenesulfonamide (*Table 4*, *Entry 14*):** Pale yellow solid; mp. 97–99°C (lit.[6] 102–104°C); IR (KBr): 2947, 2253, 1599, 1337, 1159 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 2.43 (s, 3H, $ArCH_3$), 2.69 (t, $J = 6.9$ Hz, 4H, $2NCCH_2$), 3.47 (t, $J = 6.9$ Hz, 4H, $2NCH_2$), 7.30 (d, $J = 7.9$ Hz, 2H, H_3 and H_5 of the aromatic ring), 7.72 (d, $J = 7.9$ Hz, 2H, H_2 and H_6 of the aromatic ring); ^{13}C NMR ($CDCl_3$): δ 18.9, 22.1, 45.6, 117.6, 127.2, 129.8, 135.3, 144.4; MS (m/z): 277 (M^+).

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