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Thiourea/NCBSI/HCl System: Telescoping Alkyl Halide to Alkyl Sulfonyl Chloride by Recyclable *N*-Chloro-*N*-(phenylsulfonyl) benzene Sulfonamide (NCBSI)

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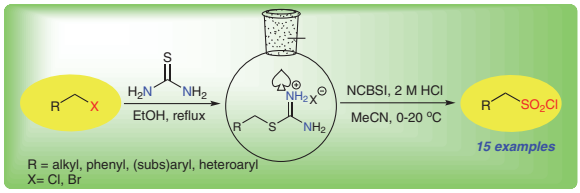

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Abstract A convenient and efficient method to synthesize diverse alkyl sulfonyl chlorides through *N*-chloro-*N*-(phenylsulfonyl)benzene sulfonamide (NCBSI)-mediated oxidative chlorosulfonation of *S*-alkyl isothiouronium salts obtained from alkyl chlorides is presented. Synthesizing structurally diverse alkyl sulfonyl chloride in moderate to excellent yields up to 98% from alkyl halide was achieved *via* easy formation of *S*-alkyl isothiouronium salts using inexpensive thiourea. The mild reaction conditions and broad substrate scope make this method attractive for alkylsulfonyl chloride syntheses.

Key words alkyl sulfonyl chlorides, *N*-chloro-*N*-(phenylsulfonyl)benzene sulfonamide, *S*-alkyl isothiouronium salts, alkyl halides, chlorosulfonation

Sulfonyl chlorides are essential synthetic intermediates that are widely used as building blocks in synthetic and medicinal chemistry.[1,2] Sulfonamides are the primary derivative of sulfonyl chloride, commonly featured in anticonvulsants, selective serotonin receptor agonists, and cytosolic phospholipase $A_2\alpha$ inhibitors (Figure 1).[3,4] Sulfonyl chlorides are frequently used as building blocks in medicinal chemistry because sulfonyl chloride reacts with heterocyclic amines to form complex sulfonamides.[5] Concerning their significance, sulfonyl chlorides have received increasing interest in the past centuries. Several research studies on the synthesis of sulfonyl chlorides from sulfides,[6] sulfonic acids,[7] or their sodium salts[7] or alkyl halides[8,9] have been reported using a variety of reaction procedures with different types of toxic and hazardous chlorinating reagents or by oxidative chlorination of sulfur-containing compounds such as thiols,[10,11] thioacetates,[11a] or disulfides[11f] with oxidizing reagents. The use of chlorine water[12] has been the most well-known method; however, handling chlorine is hazardous. Many methodologies have been reported for synthesizing sulfonyl chlorides by the dehydration of sulfonic acids with highly corrosive and toxic reagents, such as $POCl_3$, PCl_5, and $SOCl_2$.[13b,c]

S-Alkyl isothiouronium salts are readily synthesized and inexpensive starting materials for conversion into the subsequent alkyl sulfonyl chlorides *via* oxidative chlorosulfonation. HCl-treated silica gel and PhIO,[6] *t*-BuOCl-H_2O,[9] $NaClO_2$/HCl,[10] chlorine gas,[12] and *N*-chloro succinamide/HCl[14] have been applied in the oxidative chlorosulfonation. *N*-Chloro succinamide/HCl generates the water-soluble organic by-product succinimide, which leads to problems with isolation.

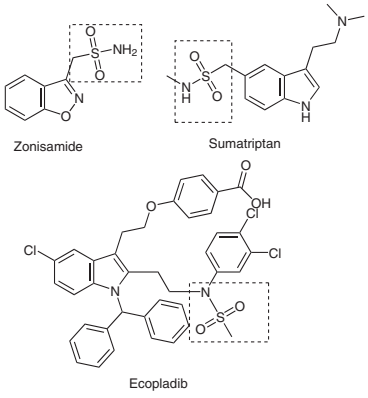
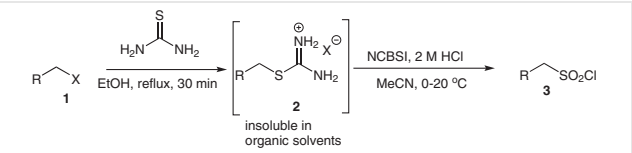


Figure 1 Structure of biologically active sulfonamide derivatives generated from sulfonyl chloride as building blocks

Recently, *N*-chloro-*N*-(phenylsulfonyl)benzene sulfonamide (NCBSI) was explored for chlorinating reactive aromatic compounds[15] and oxidation of alcohols and ethers.[15b] It is a recyclable, environmentally friendly, atom-economic, and recyclable reagent for chlorination and oxidation.

The reactivity and electrophilicity of NCBSI can be evident from its longer N–Cl bond length (1.848 Å) compared to other chlorinating reagents such as NCS, TCCA, NCSAC, and *N*-chlorophthalimide. The longer bond length is due to a strong electron pull by the two neighboring sulfonyl groups, lowering the absolute charge density on the nitrogen atom. Nitrogen thus exerts a less electron-withdrawing effect on chlorine atoms, which results in a lower absolute charge density of chlorine, which, in turn, lowers the BDE (40.36 kcal mol^{-1}).[15a] Based on the earlier study[15] NCBSI is highly reactive and results in instantaneous reaction with the advantage of by-product recyclability.

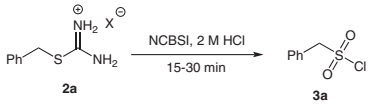
For the oxidative chlorosulfonation of *S*-alkyl isothiouronium salts, NCBSI should be an alternate, atom-economic reagent (Scheme 1). Thus, we herein report the use of thiourea/ NCBSI/ HCl as a valuable reagent system for oxidative chlorosulfonation of alkyl halides to give the corresponding sulfonyl chlorides *via* *S*-alkyl isothiouronium.



Scheme 1 Synthesis of alkane sulfonyl chlorides from *S*-alkyl isothiouronium salts via NCBSI oxidative chlorosulfonation

At the outset, we prepared *S*-benzyl isothiouronium chloride (**2a**)[16] by reacting benzyl chloride **1a** with thiourea in ethanol for 30 minutes under reflux (Table 1). Reaction optimization was commenced by reacting the model substrate **2a** with NCBSI and 2 M HCl. The oxidative chlorosulfonation was carried out in polar solvents to evaluate the solvent effect. Reacting **2a** with a suspension of NCBSI (4 equiv) in 2 M HCl with water as solvent resulted in an unsatisfactory yield (entry 1). Reaction in EtOH showed no conversion (entry 2). When MeCN was used as a solvent, an improved yield of 42% was obtained (entry 3). To our delight, further optimization by varying HCl concentrations gave 96% yield of the desired product phenylmethanesulfonyl chloride (**3a**) (entry 4).

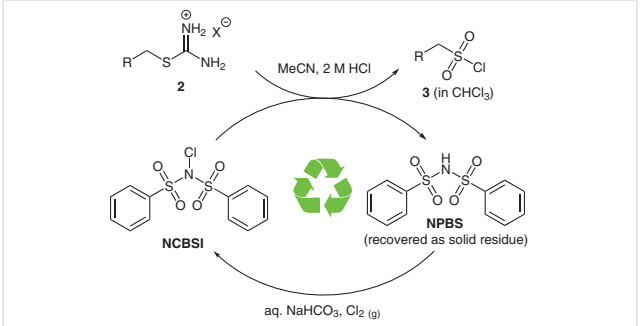
Table 1 Optimization of the Chlorosulfonation[a]



Entry	NCBSI (equiv)	Solvent	HCl	Yield (%)[b]
1	4	H_2O	1 M HCl	20
2	4	EtOH	1 M HCl	no reaction
3	4	MeCN	1 M HCl	42
4	4	MeCN	2 M HCl	96[c]

[a] Reaction conditions: S-benzyl isothiouronium salt **2a** (1.014 gm, 1 equiv), solid NCBSI (4 equiv), HCl, added sequentially to solvent (10 mL).
[b] Isolated yield.
[c] *S*-Benzyl isothiouronium salt added to a solution of NCBSI, 2 M HCl, and MeCN (10 mL), r.t.

Under the optimized conditions, the starting material **2a** was consumed completely to produce **3a**. After the reaction, acetonitrile was evaporated to obtain a mixture of the desired compound and by-product. Chloroform was added to dissolve the desired compound and the by-product as residue, which was filtered. The filtrate containing the desired compound was washed with water and sodium bicarbonate to remove traces of the by-product, and the organic layer was dried and evaporated to obtain the desired products in high yield. The recovered by-product *N*-(phenylsulfonyl)benzene sulfonamide (NPBS) from the residue could be recycled to NCBSI by treatment with sodium bicarbonate and chlorine gas in aqueous media (Scheme 2).[15a]

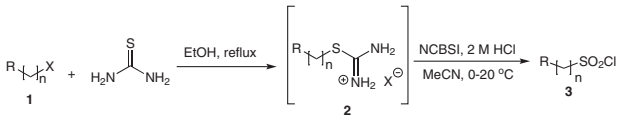


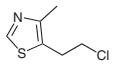
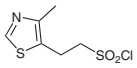
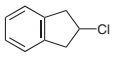
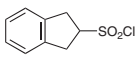
Scheme 2 Synthesis of alkyl sulfonyl chlorides by NCBSI chlorosulfonation

To demonstrate the applicability of this procedure to prepare sulfonyl chlorides, a series of sulfonyl chlorides were synthesized (Table 2). The monosubstituted S-benzyl isothiouronium salts **2a–h** were prepared in 30 minutes, while the propylphenyl did not affect reaction time (**2i**). The phenyl propyl and heterocyclic ethyl chloride required 60 minutes to form *S*-alkyl isothiouronium salts **2i** and **2j**. The aliphatic primary alkyl chlorides were converted into the corresponding S-alkyl isothiouronium salt **2k–m** in 30 minutes, while secondary and tertiary alkyl chloride derivatives required 45 minutes for conversion into **2n** and **2o**, respectively.

The time needed to convert *S*-alkyl isothiouronium salts into alkanesulfonyl chlorides varied from 15 to 60 minutes, and the yields ranged from good to excellent (Table 2). The unsubstituted and halo-substituted benzyl chlorides were converted into the corresponding sulfonyl chloride in 45 minutes overall due to the inductive effect (–I) of phenyl

Table 2 Synthesis of Structurally Diverse Alkanesulfonyl Chlorides[a]



Entry	1	1	Time (min.)[b]	Time (min.)[c]	3	Yield (%)[d]
1	C_6H_5	**1a**	30	15	$C_6H_5SO_2Cl$	97
2	4-F-C_6H_4	**1b**	30	15	4-F-$C_6H_4SO_2Cl$	94
3	4-Cl-C_6H_4	**1c**	30	15	4-Cl-$C_6H_4SO_2Cl$	96
4	4-Br-C_6H_4	**1d**	30	15	4-Br-$C_6H_4SO_2Cl$	94
5	2-I-C_6H_4	**1e**	30	15	2-I-C_6H_4 SO_2Cl	85
6	2-F-C_6H_4	**1f**	30	15	2-F-$C_6H_4SO_2Cl$	93
7	4-H_3C-C_6H_4	**1g**	30	30	4-CH_3-$C_6H_4SO_2Cl$	97
8	4-O_2N-C_6H_4	**1h**	30	25	4-O_2N-$C_6H_4SO_2Cl$	86
9	C_6H_5-$(CH_2)_3$	**1i**	60	30	C_6H_5-$(CH_2)_3SO_2Cl$	94
10		**1j**	60	45		87
11	*n*-C_3H_7	**1k**	30	60	$C_3H_7SO_2Cl$	95
12	*n*-C_4H_9	**1l**	30	60	$C_4H_9SO_2Cl$	77
13	*n*-C_5H_{11}	**1m**	30	60	$C_5H_{11}SO_2Cl$	98
14		**1n**	45	30		78
15	Ph_3C-	**1o**	45	20	Ph_3C	96

[a] Reaction conditions: Step 1: alkyl halide (1 equiv) and thiourea (1 equiv) in EtOH; Step 2: NCBSI (4 equiv), 2 M HCl, 10 mL MeCN, 0–20 °C.
[b] Reflux time .
[c] Time for oxidative chlrosulfonation.
[d] Isolated yield.

ring (**3a–d**, **3f**) with excellent yields 93 to 97%; the *ortho*-iodo-substitution product **3e** was obtained in a yield of 85%. The substrate range was expanded by introducing an electron-donating or electron-withdrawing group on the phenyl ring of benzyl chloride. An electron-releasing methyl substituent on the phenyl ring required a longer reaction time but gave **3g** in 97% yield. In contrast, an electron-withdrawing nitro-substituted phenyl ring resulted in a moderate yield of 86% with extended reaction time (**3h**). The phenylpropyl chloride (**3i**) required a little longer, although the yields were obtained at 94%, respectively.

Heterocyclic ethyl sulfonyl chlorides and fused ring sulfonyl chlorides were prepared to extend the substrate scope. The reaction time for the heterocyclic alkyl halide was longer, giving a moderate yield of 87% (**3j**). Due to the inductive effect (+I), the alkyl substrates required a much longer time but gave a good yield of 95–98% (**3k** and **3m**); however, **3l** gave a moderate yield of 77%. The secondary alkyl sulfonyl chloride required a slightly longer reaction time and gave a moderate yield of 78% (**3n**). In comparison, tertiary alkyl sulfonyl chloride was obtained with an excellent yield of 96% with a shorter reaction time (**3o**).

The proposed mechanism for synthesizing sulfonyl chloride from alkyl chloride *via* NCBSI-mediated oxidative chlorosulfonation is shown in Scheme 4. *S*-Alkyl isothiouronium salts **2** were prepared from alkyl halide and thiourea. HCl provides an aqueous acidic medium, and NCBSI is used to form alkyl sulfonyl methanimidamide salt **4**, followed by oxidation steps. Intermediate **4** reacts readily with water because of the methanimidamide salt moiety's high electrophilicity in combination with an electron-withdrawing sulfonyl group. Accordingly, intermediate **4** is converted into the corresponding sulfinic acid **5** by the water attack, elimination of halogen, proton transfer, and protonated urea elimination sequentially. Finally, sulfinic acid **5** undergoes chlorination and elimination of the hydroxyl group to give the corresponding sulfonyl chloride **3**. Regarding the significant importance and wide application of sulfonyl

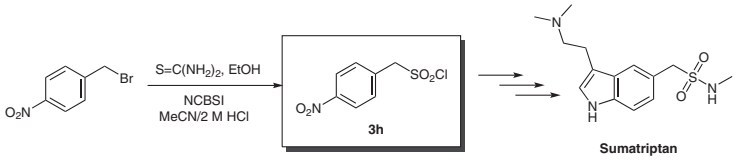
chlorides in both synthetic and pharmaceutical fields, we then directed our effort to the synthesis of intermediate p-nitrophenyl methanesulfonyl chloride which is used for synthesis of sumatriptan (Scheme 3).

In conclusion, a method was developed to synthesize alkanesulfonyl chlorides from alkyl halide in a one-pot, two-step reaction with thiourea and NCBSI. Furthermore, a one-pot conversion of alkanesulfonyl chlorides from alkyl halide has been developed. Various alkyl sulfonyl chlorides with aryl, heterocyclic, and aliphatic straight-chain compounds were synthesized in good to excellent yields by using this procedure. The key advantages of this method are the economical and readily available reagents, mild reaction conditions, excellent yields, ease of workup, and recyclability of the reagent by-product.

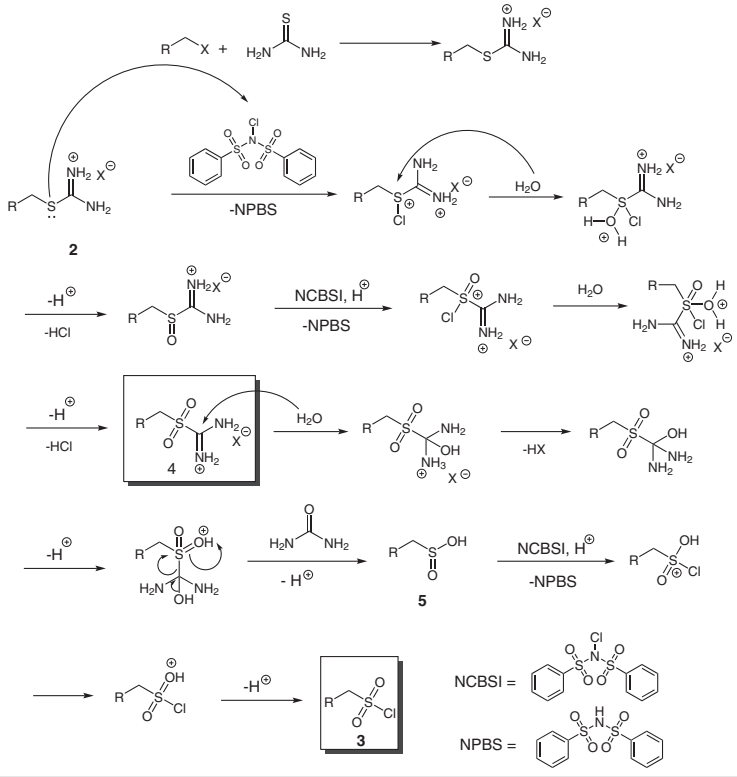
All solvents and reagents were obtained from Avra Synthesis, Spectrochem, and SD Fine Chemicals and were utilized without purification. All reactions were carried out with oven-dried glassware in a fume hood, magnetically agitated, and heated in an oil bath. The reactions were monitored by TLC on Merck silica gel G F254 plates. Melting points were recorded with an Analab ThermoCal instrument in open glass capillaries and are uncorrected. ^{1}H and $^{13}C\{^{1}H\}$ NMR spectra were recorded with an MR500 NMR spectrometer, Agilent Technologies $CDCl_3$ and DMSO-d_6 with tetramethylsilane (TMS) as the internal standard. Chemical shifts are reported in delta (δ) units in parts per million (ppm). The peak patterns are indicated as s, singlet; d, doublet; t, triplet; m, multiplet; q, quartet.

Sulfonyl Chlorides 3; General Procedure

An equimolar quantity of alkyl halide and thiourea were heated at 80 °C in EtOH (5 mL). EtOH was evaporated under vacuum and the obtained solid or viscous liquid was gradually added to a mixture of NCBSI (4 equiv), 2 M HCl (2 mL), and MeCN (10 mL) at 0–10 °C in an ice bath. The progress of the reaction was monitored by TLC. After the



Scheme 3 Application of (4-nitrophenyl)methanesulfonyl chloride to the synthesis of Sumatriptan



Scheme 4 Possible mechanism for oxidative chlorosulfonation of *S*-alkyl isothiouronium salts mediated by NCBSI

reaction reached completion, MeCN was evaporated under vacuum and chloroform (15 mL) was added to the resultant solid or suspension. The mixture was filtered and the filtrate was washed with water (15 mL) and sat. $NaHCO_3$ solution (5 mL). The organic layer was dried over Na_2SO_4 and concentrated under reduced pressure. The residue was subjected to short-column filtration (silica gel; chloroform) to give the desired products.

All the synthesized products were characterized by melting point and NMR spectroscopy.

Phenylmethanesulfonyl Chloride (3a)

Yield: 0.92 g (97%); colorless crystals; mp 92–96 °C (Lit.[17] 91–93 °C).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.45–7.35 (m, 5 H), 4.80 (s, 2 H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ = 131.30, 129.14, 126.12, 70.90.

(4-Fluorophenyl)methanesulfonyl Chloride (3b)

Yield: 0.98 g (94%); colorless crystals; mp 66–68 °C (Lit.[9] 68–69 °C).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.47 (dd, *J* = 8.5, 5.2 Hz, 2 H), 7.15 (t, *J* = 8.5 Hz, 2 H), 4.84 (s, 2 H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ = 164.88, 162.88, 133.43, 122.07, 116.46, 69.97.

Anal. Calcd: C, 40.30; H, 2.90; S, 15.37. Found: C, 41.56; H, 3.12; S, 15.31.

(4-Chlorophenyl)methanesulfonyl Chloride (3c)

Yield: 1.08 g (96%); colorless crystals; mp 92–93 °C (Lit.[18] 88–91 °C).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.40–7.33 (m, 4 H), 4.76 (s, 2 H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ = 136.74, 132.63, 129.53, 124.59, 69.97.

(4-Bromophenyl)methanesulfonyl Chloride (3d)

Yield: 1.27 g (94%); colorless crystals; mp 114–118 °C (Lit.[19] 116–118 °C).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.43–7.40 (m, 2 H), 7.20 (d, *J* = 2.5 Hz, 1 H), 7.18 (d, *J* = 1.7 Hz, 1 H), 4.46 (s, 2 H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ = 136.76, 136.42, 131.88, 130.39, 129.77, 122.45, 45.38.

(2-Iodophenyl)methanesulfonyl Chloride (3e)

Yield: 1.35 g (85%); yellowish liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.87 (d, *J* = 7.9 Hz, 1 H), 7.48 (d, *J* = 6.1 Hz, 1 H), 7.36 (t, *J* = 7.4 Hz, 1 H), 7.01 (t, *J* = 8.6 Hz, 1 H), 4.68 (s, 2 H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ = 139.88, 130.23, 128.93, 99.71, 51.15.

Anal. Calcd: C, 26.56; H, 1.91; S, 10.13. Found: C, 27.71; H, 2.33; S, 11.94.

(2-Fluorophenyl)methanesulfonyl Chloride (3f)[20]

Yield: 0.97 g (93%); colorless crystals; mp 52–54 °C (Lit.[20] 52–53.5 °C).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.44 (dt, *J* = 21.5, 6.6 Hz, 2 H), 7.16 (dt, *J* = 18.1, 8.2 Hz, 2 H), 4.89 (s, 2 H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ = 162.44, 133.03, 132.57, 124.87, 116.31, 116.14, 63.82.

Anal. Calcd: C, 40.30; H, 2.90; S, 15.37. Found: C, 40.65; H, 2.93; S, 14.89.

(4-Methylphenyl)methanesulfonyl Chloride (3g)

Yield: 0.99 g (97%); colorless crystals; mp 79–81 °C (Lit.[18] 73–75 °C).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.31 (d, *J* = 7.9 Hz, 2 H), 7.20 (t, *J* = 3.9 Hz, 2 H), 4.77 (s, 2 H), 2.34 (s, 3 H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ = 137.89, 137.35, 129.22, 127.10, 65.20, 21.12.

(4-Nitrophenyl)methanesulfonyl Chloride (3h)

Yield: 1.01 g (86%); off-white crystals; mp 84–86 °C (Lit.[21] 92–93 °C).

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.13 (d, *J* = 8.7 Hz, 2 H), 7.47 (d, *J* = 8.7 Hz, 2 H), 4.55 (s, 2 H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ = 147.73, 144.28, 129.32, 123.96, 44.50.

3-Phenylpropane-1-sulfonyl Chloride (3i)

Yield: 1.09 g (94%); clear viscous liquid.[22]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.31–7.07 (m, 5 H), 3.52–3.44 (m, 2 H), 2.88–2.68 (m, 2 H), 2.07–1.99 (m, 2 H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ = 141.88, 128.43, 125.87, 62.13, 34.22, 32.10.

2-(4-Methylthiazol-5-yl)ethane-1-sulfonyl Chloride (3j)

[CAS No. 1342688-76-9]

Yield: 0.98 g (87%); darkyellow liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 8.85 (s, 1 H), 3.61 (t, *J* = 6.8 Hz, 2 H), 3.17 (t, *J* = 6.8 Hz, 2 H), 2.39 (s, 3 H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ = 149.98, 147.63, 127.22, 43.08, 28.52, 13.38.

Anal. Calcd: C, 31.93; H, 3.57; S, 28.41; N, 6.21. Found: C, 34.24; H, 3.94; S, 29.57; N, 7.12.

Propane-1-sulfonyl Chloride (3k)

Yield: 0.68 g (95%); light yellow liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 3.63–3.54 (m, 2 H), 2.02 (tt, *J* = 14.4, 7.2 Hz, 2 H), 1.08 (t, *J* = 7.5 Hz, 3 H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ = 66.95, 18.19, 12.26.

Butane-1-sulfonyl Chloride (3l)

Yield: 0.60 g (77%); light orange liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 3.64–3.57 (m, 2 H), 2.00–1.93 (m, 2 H), 1.52–1.43 (m, 2 H), 0.94 (t, *J* = 7.4 Hz, 3 H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ = 65.22, 26.18, 20.90, 13.39.

Pentane-1-sulfonyl Chloride (3m)

Yield: 0.84 g (98%); light yellow liquid.

^{1}H NMR (500 MHz, $CDCl_3$): δ = 3.63–3.56 (m, 2 H), 2.02–1.92 (m, 2 H), 1.46–1.38 (m, 2 H), 1.37–1.29 (m, 2 H), 0.88 (t, *J* = 7.2 Hz, 3 H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ = 65.43, 29.59, 23.96, 22.00, 13.62.

2,3-Dihydro-1*H*-indene-2-sulfonyl Chloride (3n)

Yield: 0.84 g (78%); colorless oil.[23]

^{1}H NMR (500 MHz, $CDCl_3$): δ = 7.23–7.10 (m, 4 H), 4.71–4.66 (m, 1 H), 3.38 (dt, *J* = 31.8, 15.9 Hz, 2 H), 3.21–3.10 (m, 2 H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ = 140.82, 126.65, 124.99, 73.15, 42.62.

Anal. Calcd: C, 49.89; H, 4.19; S, 14.80. Found: C, 47.96; H, 4.36; S, 13.53.

Triphenylmethanesulfonyl Chloride (3o)

Yield: 1.65 g (96%); colorless crystals.

^1H NMR (500 MHz, $CDCl_3$): δ = 7.37–7.24 (m, 15 H).

$^{13}C\{^1H\}$ NMR (126 MHz, $CDCl_3$): δ = 146.85, 127.94, 127.26, 82.04.

Anal. Calcd: C, 66.56; H, 4.41; S, 9.35. Found: C, 62.32; H, 4.36; S, 8.52

Recovery of Reagent

To recover the *N*-(phenylsulfonyl)benzene sulfonamide, a starting material of NCBSI, a gram-scale model reaction was performed. The residue from the reaction was collected and washed with ice-cold water and dried in an oven at 65 °C to afford 77.8 % of *N*-(phenylsulfonyl)benzene sulfonamide, This can be reused for the preparation of NCBSI. The recovered compound may contain a mixture of *N*-(phenylsulfonyl)benzene sulfonamide and NCBSI. The *N*-(phenylsulfonyl)benzene sulfonamide was purified by recrystallization and its identity was confirmed by NMR analysis.

Conflict of Interest

The authors declare no conflict of interest.

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Supporting Information

Supporting information for this article is available online at https://doi.org/10.1055/a-2360-9229.

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