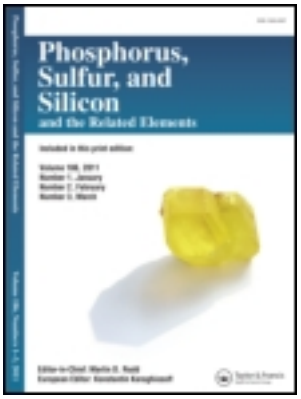

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Diverse Reactions of Sulfonyl Chlorides and Cyclic Imines

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DIVERSE REACTIONS OF SULFONYL CHLORIDES AND CYCLIC IMINES

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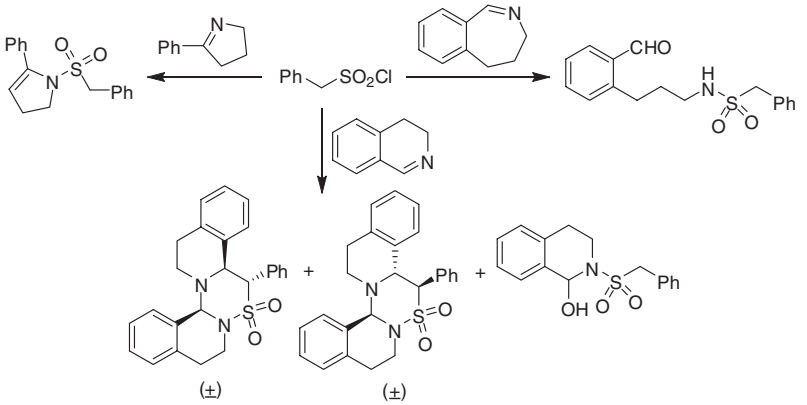
GRAPHICAL ABSTRACT



Abstract *Although alkanesulfonyl chlorides react with linear imines to give rise to β-sultam derivatives, in this study, they were reacted with various cyclic imines, including 1-pyrroline, oxazoline, 5,6-dihydro-4H-oxazines and thiazines, 4,5-dihydro-3H-benzo[c]azepine, and 3,4-dihydroisoquinoline, to produce diverse products instead of β-sultam derivatives. The results indicate that alkanesulfonyl chlorides react with cyclic imines to generate N-alkanesulfonyl cyclic iminium ions, which are attacked by nucleophiles, such as water and chloride anion, in the reaction systems, affording addition products. The iminium intermediates cannot undergo a ring closure to form β-sultam derivatives. Arenesulfonyl chlorides showed similar behavior when they reacted with cyclic imines. The scope and limitation of the reaction between sulfonyl chlorides and imines were investigated.*

Keywords Alkanesulfonyl chloride; arenesulfonyl chloride; imine; β-sultam; sulfonyl chloride

INTRODUCTION

β-Sultams (1,2-thiazetidine 1,1-dioxides) have been considered as important sulfur analogs of β-lactams, and are key skeletons in pharmaceutical chemistry and crucial

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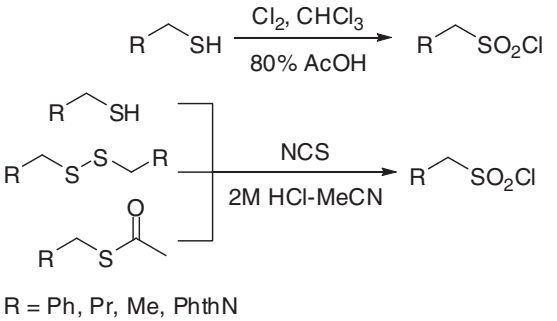
intermediates in synthetic organic chemistry.[1,2] Several synthetic methods for β-sultams have been developed.[3] They have been synthesized via the reaction of alkanesulfonyl chlorides and imines[3–6] and via the reaction of alkylaminosulfonyl chlorides and electron-rich olefins[7,8] through β-aminoalkanesulfonyl halides,[3,8,9] which were prepared from the Michael addition of amines to alkyl α,β-alkenesulfonates, hydrolysis, and subsequent halogenation,[8] or from nucleophilic ring opening of thiiranes with amines or aziridines with hydrogen sulfide and subsequent oxidation with chlorine or *N*-chlorosuccimide (NCS),[7] via cyclization of β-hydroxyalkanesulfonamides through their methanesulfonates[10] or via cyclization of α-haloalkanesulfonamides in the presence of bases.[11]

Since most of efficient β-lactam antibiotics are polycyclic compounds containing a fused β-lactam moiety, polycyclic compounds containing a fused β-sultam moiety are also of pharmaceutical interest.[2,9,12] β-sultam-fused polycycles have only been prepared either by cyclocondensation of 2-aminoethanesulfonyl chloride hydrochloride derivatives in ethyl acetate in the presence of potassium carbonate as base[9] or by cyclization of amines with vinylsulfonyl fluoride derivatives via tandem Michael addition and aminolysis in multiple steps with limitation of the structural diversity of products.[8] We investigated the reactivity of various alkanesulfonyl chlorides and cyclic imines and present herein our results.

RESULTS AND DISCUSSION

Synthesis of Alkanesulfonyl Chlorides

Alkanesulfonyl chlorides can be prepared from the corresponding alkanethiols, alkyl disulfides, and alkylthio acetates via chlorine[13,14] or NCS[15] oxidation (Scheme 1). Benzylthiol and butanethiol were oxidized with chlorine and NCS, respectively. The results indicate that the chlorine oxidation gave rise to higher yields of products than the NCS oxidation (Table 1, entries 1–4). However, alkanesulfinyl chlorides were observed (by NMR analysis for the products) under chlorine oxidation when insufficient chlorine was bubbled in. It is inconvenient to control the amount of chlorine gas in laboratory and excessive chlorine gas also pollutes the environment. The NCS oxidation is a convenient and maneuverable procedure despite that it produced lower yields of the products. Thus, ethyl disulfide and *N*-acetylthiomethylphthalimide were oxidized with NCS to afford ethanesulfonyl and phthalimidomethanesulfonyl chlorides, respectively, in satisfactory yields (Table 1, entries 5–6).



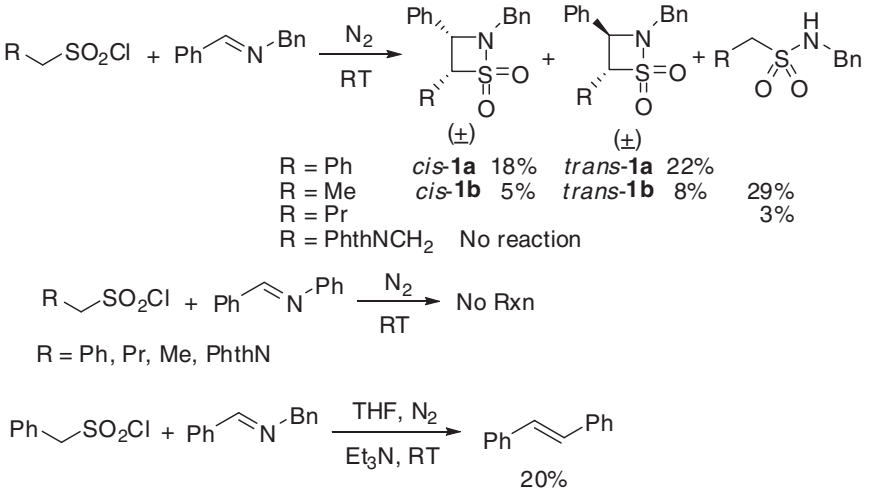
Scheme 1 Synthesis of alkanesulfonyl chlorides.

Table 1 Synthesis of alkanesulfonyl chlorides

Entry	Thiol/disulfide/thioester	Oxidant	Product	Yield (%)
1	BnSH	Cl_2	$BnSO_2Cl$[13]	98.5
2	BuSH	Cl_2	$BuSO_2Cl$[14]	80
3	BnSH	NCS	$BnSO_2Cl$	67.6
4	BuSH	NCS	$BuSO_2Cl$	67.6
5	EtSSEt	NCS	$EtSO_2Cl$[15]	47.9
6	$PhthNCH_2SAc$	NCS	$PhthNCH_2SO_2Cl$[16]	48.5

Reactions of Sulfonyl Chlorides and Imines

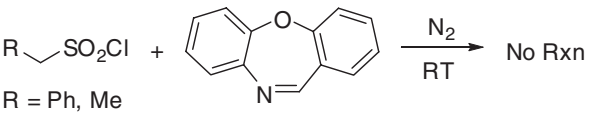
Although it has been reported that alkanesulfonyl chlorides react with linear imines to yield β-sultam derivatives, only imines generated from alkylamines and arylmethylamines were used in the reported examples.[3–6] To investigate the generality of the reaction, we conducted the reactions of alkanesulfonyl chlorides with two equivalents of *N*-benzylidenebenzylamine according to the reported procedure.[3–6] The reactions of benzylsulfonyl and ethanesulfonyl chlorides produced the corresponding *cis*- and *trans*-2-benzyl-3,4-diphenyl-β-sultams (*cis*-**1a** and *trans*-**1a**) or *cis*- and *trans*-2-benzyl-4-methyl-3-phenyl-β-sultams (*cis*-**1b** and *trans*-**1b**), respectively, in lower yields, as reported.[4] In the reaction of ethanesulfonyl chloride, *N*-benzyl-ethanesulfonamide was also obtained, with a yield of 39%. *N*-Benzyl-butanesulfonamide was obtained as sole product, with a 3% yield, in the reaction of butanesulfonyl chloride and *N*-benzylidenebenzylamine. However, no reaction occurred between phthalimidomethanesulfonyl chloride and *N*-benzylidenebenzylamine (Scheme 2). We also conducted the reactions of all prepared alkanesulfonyl chlorides with *N*-benzylideneaniline. No reaction occurred in each of the cases (Scheme 2). We also attempted the reaction of benzylsulfonyl chloride with one equivalent of *N*-benzylideneaniline and benzylamine, respectively, in the presence of triethylamine (as the Staudinger reaction of acyl chlorides and imines).[17] *Trans*-stilbene was



Scheme 2 Reactions of alkanesulfonyl chlorides with linear imines.

obtained as sole product instead of the desired β-sultams (Scheme 2). Juzo et al. proposed the formation mechanism of *trans*-stilbene.[18] On the basis of the reported results[3–6] and our findings, we can conclude that alkanesulfonyl chlorides can only react with *N*-alkylimines to yield β-sultams, with the excessive imines themselves acting as bases to dehydrochlorinate.

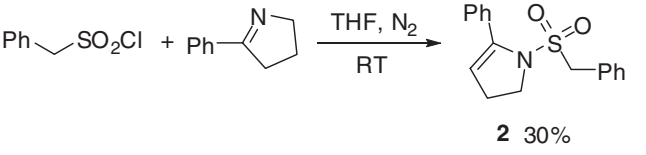
Since no example on the reaction of alkanesulfonyl chlorides and cyclic imines has been reported till now, we first attempted to react benzylsulfonyl and ethanesulfonyl chloride with dibenzo[*b*,*f*][1,4]oxazepine, a cyclic imine with an *N*-aryl group. Like linear *N*-aryl imines, no reaction occurred. Both linear and cyclic *N*-arylimines cannot react with alkanesulfonyl chloride, possibly because *N*-arylimines show weaker basicity than *N*-alkylimines and cannot dehydrochlorinate from alkanesulfonyl acid derivatives (Scheme 3).



Scheme 3 Attempted reaction of alkanesulfonyl chlorides with dibenzo[*b*,*f*][1,4]oxazepine, a cyclic imine with an *N*-aryl group.

In a further investigation, we only applied cyclic imines with *N*-alkyl groups. A series of cyclic *N*-alkyl-imines, including 1-pyrroline, oxazoline, 4,5-dihydro-3*H*-dihydrooxazines, thiazines, 4,5-dihydro-3*H*-benzo[*c*]azepine, and 3,4-dihydroisoquinoline, was synthesized according to the reported procedure and reacted with alkanesulfonyl chlorides. Diverse results were obtained.

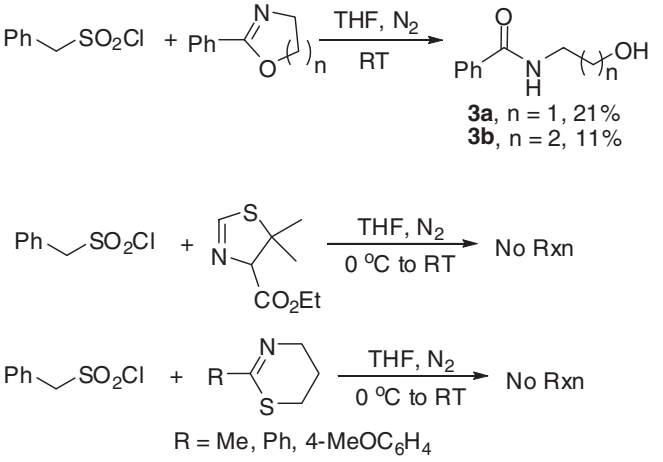
2-Phenyl-1-pyrroline reacted with benzylsulfonyl chloride to give 1-benzylsulfonyl-2-phenyl-2-pyrroline (**2**) via *N*-benzylsulfonylation and the double bond shift (Scheme 4). The results indicate that the acidity of the hydrogen atom in the C3 atom of the pyrrolinium is stronger than that in the Cα of the *N*-benzylsulfonyl group in the 1-benzylsulfonyl-2-phenyl-1-pyrrolinium intermediate, generated from 2-phenyl-1-pyrroline and benzylsulfonyl chloride.



Scheme 4 Reaction of benzylsulfonyl chloride with 2-phenyl-1-pyrroline.

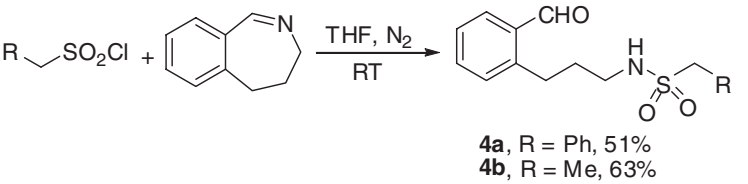
Interestingly, 2-phenyloxazoline and 2-phenyl-5,6-dihydro-4*H*-1,3-oxazine reacted with benzylsulfonyl chloride to give rise to *N*-(2-hydroxyethyl)benzamide (**3a**) and *N*-(3-hydroxypropyl)benzamide (**3b**), respectively. It is obviously different from the reaction with alkanoyl chlorides, in which *N*-(2-hydroxyethyl)- and *N*-(3-hydroxypropyl)alkanamides were obtained rather than the corresponding benzamides.[19] In the current cases, benzamides were generated rather than benzylsulfonamides, revealing that *N*-benzylsulfonylation did not occur and the corresponding benzamides yielded via sulfonic acid-catalyzed hydrolysis during workup. The sulfonic acid was formed from sulfonyl chloride reacting with

water during workup (Scheme 5). However, no reaction was observed when ethyl 5,5-dimethylthiazolidine-4-carboxylate and 5,6-dihydro-4*H*-1,3-thiazines reacted with benzylsulfonyl chloride (Scheme 5).



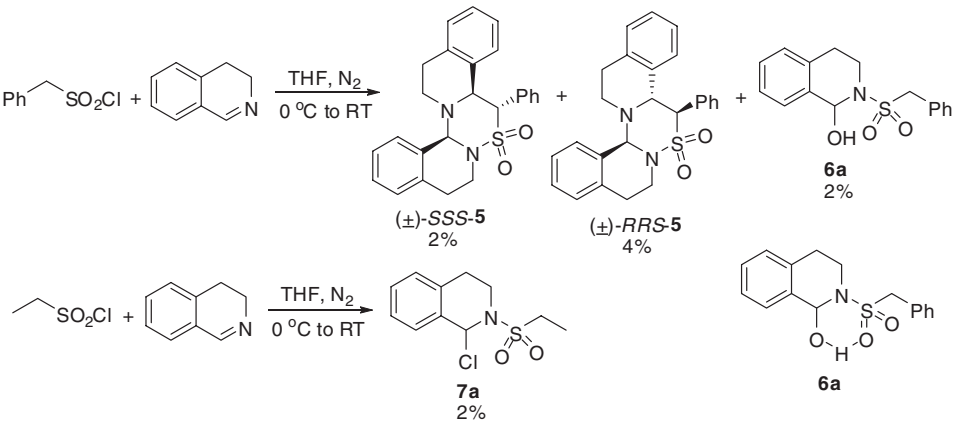
Scheme 5 Reaction of benzylsulfonyl chloride with oxazoline, thiazoline, 5,6-dihydro-4*H*-1,3-oxazine and thiazines.

4,5-Dihydro-3*H*-benzo[*c*]azepine reacted with alkanesulfonyl chloride to give rise to *N*-[3-(2-formylphenyl)propyl]alkanesulfonamides (**4**) via *N*-alkanesulfonylation and the further hydrolysis during workup as the reaction of oxazoline with acyl chlorides[19] (Scheme 6).



Scheme 6 Reaction of alkanesulfonyl chlorides and 4,5-dihydro-3*H*-benzo[*c*]azepine.

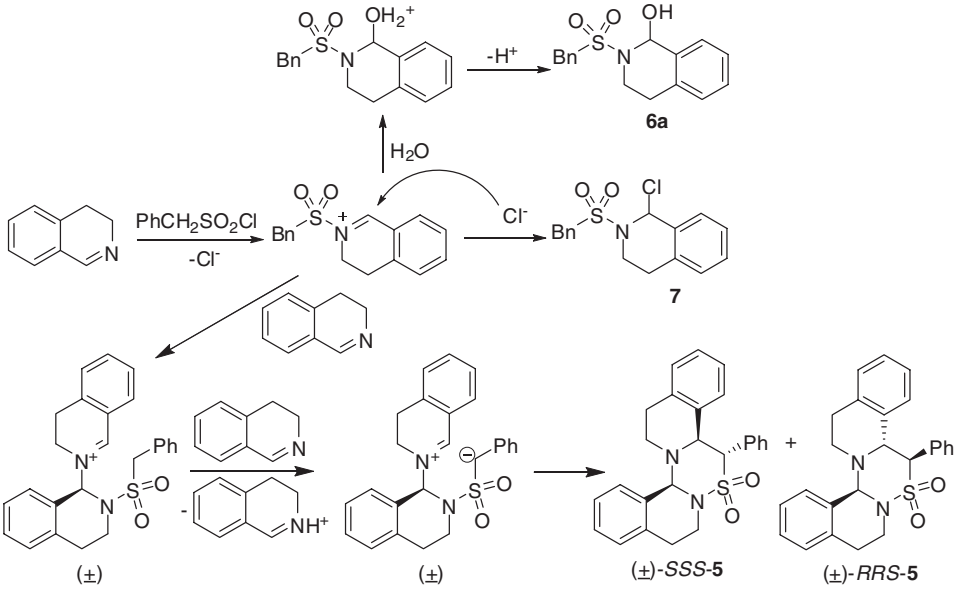
The reaction of 3,4-dihydroisoquinoline with alkanesulfonyl chlorides led to complex products (Scheme 7). It gave rise to *rel*-(8*S*,9a*S*,12a*S*)- and *rel*-(8*R*,9a*R*,12a*S*)-9-phenyl-5,6,9,9a,14,15-hexahydro-16a*H*-diisoquinolino[2,1-*b*:2′,1′-*d*][1,2,4]thiadiazine-8,8-dioxides (*SSS*-**5** and *RRS*-**5**), a formal [2 + 2 + 2] cycloadduct generated from benzylsulfonyl chloride and two molecules of 3,4-dihydroisoquinoline and 2-(benzylsulfonyl)-1,2,3,4-tetrahydroisoquinolin-1-ol (**6**), generated via benzylsulfonylation and subsequent addition with water and deprotonation during workup, in very low yields when it reacted with benzylsulfonyl chloride, while it produced 1-chloro-2-(ethylsulfonyl)-1,2,3,4-tetrahydroisoquinoline (**7**) as sole product, generated via ethanesulfonylation and subsequent addition with chloride anion in the reaction system, when it reacted with ethanesulfonyl chloride. The results indicate that the *N*-alkanesulfonyliminium ions generated in the reactions could not undergo a ring closure to produce β-sultams, but



Scheme 7 Reaction of alkanesulfonyl chlorides with 3,4-dihydroisoquinoline.

underwent an addition with another molecule of 3,4-dihydroisoquinoline and subsequent ring closure to generate formal [2 + 2 + 2] products, or underwent nucleophilic additions with chloride anion in the reaction system or with water during workup, affording 1-chloro-2-alkylsulfonyl- 1,2,3,4-tetrahydroisoquinoline (**6**) or 2-alkylsulfonyl-1,2,3,4-tetrahydroisoquinolin-1-ol (**7**), respectively (Scheme 8). For linear and other cyclic imines, the water addition to the *N*-sulfonylated iminium ions resulted in the hydrolysis of the iminium ions to afford *N*-alkylsulfonamide derivatives (Schemes 2 and 6).

The structures of products **5** were determined via ^{1}H and ^{13}C NMR, and mass spectra and were confirmed by an X-ray structure analysis. On comparison with the corresponding



Scheme 8 Mechanism of in the reaction of alkanesulfonyl chlorides with 3,4-dihydroisoquinoline.

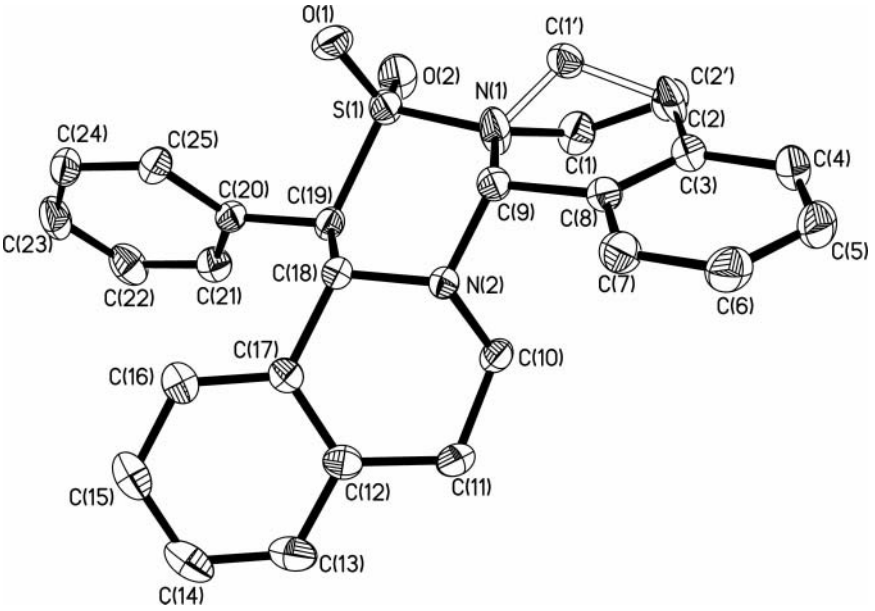


Figure 1 ORTEP structure of compound (±)-*SSS*-**5**.

desired β-sultam, five more aliphatic protons and four more aromatic protons were observed in their ^{1}H spectra and three more aliphatic carbons and four or five more aromatic carbons were found in their ^{13}C NMR spectra. Their high-resolution mass spectra indicated that the products were composed of one molecule of PhCH=SO_2 and two molecules of 3,4-dihydroisoquinoline. Referring to the formation mechanism of the products from one molecule of ketene and two molecules of imines,[20–22] the structures of products **5** were suggested as 9-phenyl-5,6,9,9a,14,15-hexahydro-16a*H*-diisoquinolino[2,1-*b*:2′,1′-*d*][1,2,4]thiadiazine-8,8-dioxides with *rel*-(8*S*,9a*S*,12a*S*)- and *rel*-(8*R*,9a*R*,12a*S*)- configurations because the vicinal CH—CH group shows coupling constants of 10.4 and 9.6 Hz, respectively, demonstrating that these two protons exhibit the *trans*-position in the 1,2,4-thiadiazine ring. It was assumed that (±)-*SSS*-**5** was generated in a lower yield (2%) than (±)-*RRS*-**5** (4%) due to steric hindrance in the ring closure because the formation of the 1,3-*anti*-structure in (±)-*RRS*-**5** is more favorable than that of the 1,3-*syn*-structure in (±)-*SSS*-**5** (Scheme 8). The structure of (±)-*SSS*-**5** was further confirmed by X-ray structural analysis (Figure 1).

The structure of product **6** was identified via ^{1}H and ^{13}C NMR, and mass spectra. Its ^{13}C NMR spectrum illustrated that was composed of benzylsulfonyl and 3,4-dihydroisoquinoline. Its ^{1}H NMR spectrum indicated that 3,4-dihydroisoquinoline was benzylsulfonylated because a singlet peak at 4.35 (2H) and nine aromatic protons were observed. Additionally, two doublet peaks at 2.94 (1H) and 6.02 (1H), respectively, with a coupling constant $J = 5.3$ Hz were observed and the peak at 2.94 disappeared after addition of a drop of D_2O, revealing that it was due to OH and that the peak at 6.02 was connected with an aromatic carbon, an oxygen, and a nitrogen atom. The mass spectrum also supports the structure. After identification of the structure of product **6**, it is very easy to determine the structure of product **7** because a peak at 6.74 (s, 1 H) in its ^{1}H NMR is connected with

Table 2 Selected bond lengths (Å)

Bond	Distance	Bond	Distance
S1−O2	1.433(2)	C1−C2	1.516(1)
S1−O1	1.439(2)	C1′−C2′	1.534(1)
S1−N1	1.648(2)	C3−C8	1.397(2)
S1−C19	1.797(2)	C8−C9	1.517(2)
N1−C1	1.372(2)	C10−C11	1.517(2)
N1−C1′	1.395(2)	C11−C12	1.506(3)
N1−C9	1.496(2)	C12−C17	1.393(2)
N2−C9	1.450(2)	C17−C18	1.519(2)
N2−C18	1.465(2)	C18−C19	1.574(2)
N2−C10	1.472(2)	C19−C20	1.511(2)

an aromatic carbon, a nitrogen, and a chlorine atom. The mass spectrum also supports the suggested structure.

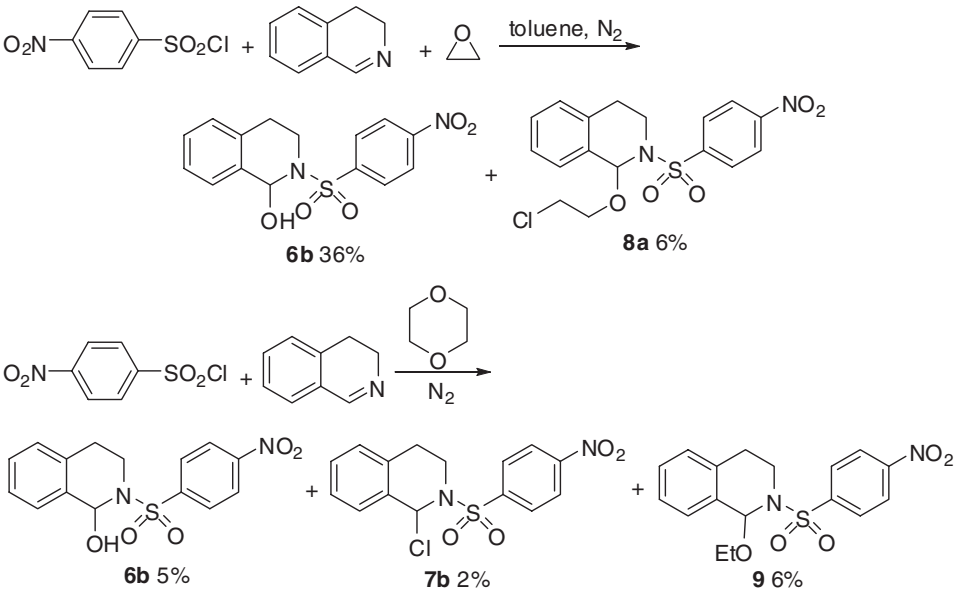
It is well known that hemiacetals and *N*-hemiacetals are generally unstable in acidic aqueous solution. Although product **6a** possesses a structural feature of *N*-sulfonylated *N*-hemiacetals, it is still stable due to the intramolecular hydrogen bond between the hydroxyl group and the oxygen atom in the sulfonyl group. A similar phenomenon was observed previously for the oxidation of 2-(2-methoxyphenyl)-4-phenyl-2,3-dihydro-1*H*-1,5-benzodiazepine with *m*-chloroperbenzoic acid.[23]

A crystal ($0.53 \times 0.46 \times 0.42$ mm^3) of compound (±)-*SSS*-**5** ($C_{25}H_{24}N_2O_2S$, $Mr = 416.52$) was crystallized from a mixture of ethyl acetate and petroleum ether (60–90°C). It crystallizes in the monoclinic space group $P2_1/n$, with cell dimensions $a = 0.\ 9852(2)$ nm, $b = 1.5942(4)$ nm, $c = 1.3118(3)$ nm, $\alpha = 90°$, $\beta = 95.199(3)°$, $\gamma = 90°$, $V = 2.0519(8)$ nm^3, $Z = 4$, $D_c = 1.348$ mg·m^{-3}, $F(000) = 880$, and $\mu = 18.3$ cm^{-1}. The structure was solved by direct methods and refined by full-matrix least squares. The final discrepancy factor is 0.0369 for 5428 observed reflections. The selected bond lengths and bond angles are compiled in Tables 2 and 3.

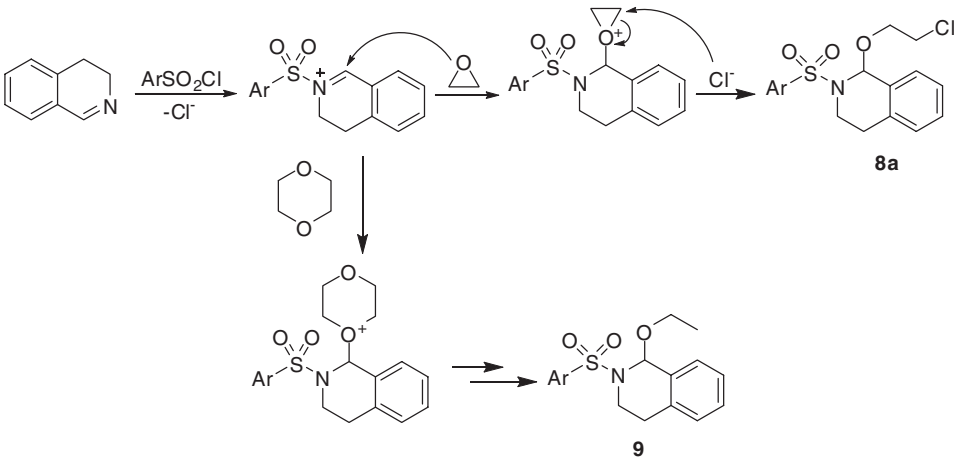
Table 3 Selected bond angles (°)

Angle	(°)	Angle	(°)
O2−S1−O1	118.3(1)	C18−N2−C10	112.6(1)
O2−S1−N1	108.1(1)	N1−C1−C2	114.3(5)
O1−S1−N1	109.0(1)	N1−C1′−C2′	110.4(4)
O2−S1−C19	110.1(1)	C3−C2′−C1′	113.9(6)
O1−S1−C19	108.1(1)	N2−C9−N1	112.6(1)
N1−S1−C19	102.1(1)	N2−C9−C8	110.7(1)
C1−N1−C1′	46.8(2)	N1−C9−C8	111.0(1)
C1−N1−C9	120.1(2)	N2−C18−C17	110.3(1)
C1′−N1−C9	122.3(2)	N2−C18−C19	111.4(1)
C1−N1−S1	125.3(2)	C17−C18−C19	111.4(1)
C1′−N1−S1	114.5(2)	C20−C19−C18	115.2(1)
C9−N1−S1	112.1(1)	C20−C19−S1	111.0(1)
C9−N2−C18	112.3(1)	C18−C19−S1	107.9(1)
C9−N2−C10	116.1(1)	C3−C2−C1	109.9(6)

From the above reactions, we found that the *N*-sulfonylated iminum ions can undergo addition reactions with weak nucleophiles, such as water and chloride anions. To explore the reactivity of *N*-alkanesulfonyl-3,4-dihydroisoquinolinium ions with weak nucleophiles, we conducted the reactions of 4-nitrobenzenesulfonyl chloride and 3,4-dihydroisoquinoline in the presence of weak nucleophilic ether solvents or additives. We used the arenesulfonyl chloride instead of alkanesulfonyl chlorides to avoid the formation of cyclization products for convenient purification of addition products. The reaction of 4-nitrobenzenesulfonyl chloride and 3,4-dihydroisoquinoline in toluene with strained oxirane as an additive gave rise to *N*-(4-nitrobenzenesulfonyl)-1,2,3,4-tetrahydroisoquinolin-1-ol (**6b**) and 1-(2-chloroethoxy)-*N*-(4-nitrobenzenesulfonyl)- 1,2,3,4-tetrahydroisoquinoline (**8a**). The yield of the product of water addition (**6b)** was indeed improved to 36% here. The yield of **6b** is higher than that of **8a**, possibly because oxirane is more bulky than water in nucleophilic attack. The reaction of 4-nitrobenzenesulfonyl chloride and 3,4-dihydroisoquinoline in 1,4-dioxane generated *N*-(4-nitrobenzenesulfonyl)-1,2,3,4-tetrahydroisoquinolin-1-ol (**6b**) and 1-chloro-*N*-(4-nitrobenzenesulfonyl)-1,2,3,4-tetrahydroisoquinoline (**7b**), and 1-ethoxy-*N*-(4-nitrobenzenesulfonyl)-1,2,3,4-tetrahydroisoquinoline (**9**) as well. The mechanism of the formation of product **9** is not clear now (Scheme 9). 1-(2-Chloroethoxy)-*N*- (4-nitrobenzenesulfonyl)-1,2,3,4-tetrahydroisoquinoline (**8a**) was generated via arenesulfonylation and oxirane nucleophilic addition to the iminium intermediate, followed by nucleophilic ring opening of the oxirane ring in the generated intermediate with chloride (Scheme 10).



Scheme 9 Reaction of arenesulfonyl chlorides with 3,4-dihydroisoquinoline in the presence of weak nucleophilic ethers.



Scheme 10 Mechanism of the reaction of arenesulfonyl chlorides with 3,4-dihydroisoquinoline in the presence of weak nucleophilic ethers.

CONCLUSION

In conclusion, the scope and limitation of the reaction of imines with sulfonyl chlorides were evaluated. The results indicate that only acyclic *N*-alkylimines can react with alkanesulfonyl chlorides to afford β-sultam derivatives. Reactions of several alkanesulfonyl chlorides and various cyclic imines were investigated. The reactions studied cannot produce the corresponding β-sultam product, instead of the double bond shifted, ring-opened, nucleophilic addition products in low yields. The results reveal that it is hard to prepare fused polycyclic β-sultam derivatives from alkanesulfonyl chlorides and cyclic imines via cycloaddition.

EXPERIMENTAL

General

Melting points were obtained on a Yanaco MP-500 melting point apparatus and are uncorrected. The ^{1}H NMR and ^{13}C NMR spectra were recorded on Varian Mercury 200, Varian Mercury 300 Plus, or Bruker 400 or 600 spectrometer with TMS as an internal standard in $CDCl_3$ solution and the chemical shifts (δ) are reported in parts per million (ppm). The IR spectra (KBr pellets, ν [cm^{-1}]) were taken on a Nicolet 5700 FTIR spectrometer. HRMS measurements were carried out on an Agilent LC/MSD TOF mass spectrometer. TLC separations were performed on silica gel GF_{254} plates, and the plates were visualized with UV light. Analytic data of all synthetic alkanesulfonyl chlorides are identical to those reported.[13–16] All cyclic imines (pyrroline,[24] oxazoline,[18] 5,6-dihydro-4*H*-oxazine[25] and thiazines,[26] 3,4-dihydroisoquinoline,[27] 4,5-dihydro-3*H*-benzo[*c*]azepine,[28] and dibenzo[*b*,*f*][1,4]oxazepine[29]) were synthesized according to reported methods. PE and EA are abbreviations for petroleum ether (60–90°C) and ethyl acetate, respectively.

Reaction of Alkanesulfonyl Chlorides with Imines: General Procedure

To a solution of an imine (10 mmol) in anhydrous THF (10 mL) was added dropwise a solution of an alkanesulfonyl chloride (5 mmol) in anhydrous THF (10 mL) in an ice-water bath (for the reaction of ethanesulfonyl chloride with benzylidene benzylamine without solvent). After addition, the resulting mixture was further stirred in the ice-water bath for 24 h and further at r.t. for 48 h. After the filtration of salts and removal of solvent, the residue was purified via silica gel column chromatography (PE/EA 1:1 to 10:1, v/v) to afford colorless crystals.

(±)-*cis*-2-Benzyl-3,4-diphenyl-β-sultam (*cis*-1a). Colorless crystals, 0.33 g (18%). mp 156–157°C.[4] mp 156–157°C. $R_f = 0.30$ (PE/EA = 3:2, v/v). ^{1}H NMR (200 MHz) δ: 4.30 (d, $J = 14.2$ Hz, 1H, CH_2), 4.54 (d, $J = 14.4$ Hz, 1H, CH_2), 4.87 (d, $J = 8.2$ Hz, 1H, NCH), 5.73 (d, $J = 8.2$ Hz, 1H, SCH), 7.09 Å) 7.49 (m, 15 H, ArH).

(±)-*trans*-2-Benzyl-3,4-diphenyl-β-sultam (*trans*-1a). Colorless crystals, 0.40 g (22%); mp 98–100°C.[4] mp 98–100°C. $R_f = 0.60$ (PE/EA = 3:2, v/v). ^{1}H NMR (200 MHz) δ: 4.23 (d, $J = 14.4$ Hz, 1H, CH_2), 4.3 (d, $J = 14.4$ Hz, 1H, CH_2), 4.36 (d, $J = 6$ Hz, 1H, NCH), 5.22 (d, $J = 6$ Hz, 1H, SCH), 7.31–7.51 (m, 15 H, ArH).

(±)-*cis*-2-Benzyl-4-methyl-3-phenyl-β-sultam (*cis*-1b). Colorless crystals, 21 mg (5%); mp 60–62°C. $R_f = 0.50$ (PE: EA = 5:1, v/v). IR: 1353 (S=O), 1140 (S=O). ^{1}H NMR (300 MHz) δ: 1.09 (d, $J = 7.2$ Hz, 3H, CH_3), 4.17 (d, $J = 14.4$ Hz, 1H, CH_2), 4.35 (d, $J = 14.4$ Hz, 1H, CH_2), 4.49 (d, $J = 8.1$ Hz, 1H, CH), 4.35 (dt, $J = 7.2$, 8.1 Hz, 1H, CH), 7.27–7.36 (m, 10H, ArH). HRMS (ESI) *m/z* for $C_{16}H_{18}NO_2S^+$ $[M + H]^+$: Calc., 288.1053; Found, 288.1063.

(±)-*trans*-2-Benzyl-4-methyl-3-phenyl-β-sultam (*trans*-1b). Colorless crystals, 16 mg (8%); mp 108–110°C. $R_f = 0.70$ (PE: EA = 5:1, v/v). IR: 1324 (S=O), 1156 (S=O). ^{1}H NMR (300 MHz) δ: 1.56 (d, $J = 6.9$ Hz, 3H, CH_3), 3.69 (d, $J = 6.3$ Hz, 1H, CH), 4.06 (d, $J = 14.1$ Hz, 1H, CH_2), 4.16 (dt, $J = 6.9$, 6.3 Hz, 1H, CH), 4.28 (d, $J = 14.1$ Hz, 1H, CH_2), 7.31–7.51 (m, 10 ArH). HRMS (ESI) *m/z* for $C_{16}H_{18}NO_2S^+$ $[M + H]^+$: Calc., 288.1053; Found, 288.1063.

***N*-Benzyl-ethanesulfonamide**. Colorless crystals, 0.58 g (29%); mp 60–62°C.[30] mp 56–58°C. ^{1}H NMR (200 MHz) δ: 1.33 (t, $J = 7.5$ Hz, 3H, CH_3), 2.97 (q, $J = 7.5$ Hz, 2H, CH_2), 4.31 (d, $J = 4.0$ Hz, 2H, NCH_2), 4.52 (bs, 1H, NH), 7.33–7.37 (m, 5H, ArH).

***N*-Benzyl-butanesulfonamide**. Colorless crystals, 0.34 g (3%); mp 72–74°C.[31] mp 72–74°C. ^{1}H NMR (200 MHz) δ: 0.87 (t, $J = 7.5$ Hz, 3H, CH_3), 1.38 (m, 2H, CH_2), 1.72 (m, 2H, CH_2N), 2.91 (t, $J = 7.5$ Hz, 2H, CH_2S), 4.31 (d, $J = 4.0$ Hz, 2H, NCH_2), 4.57 (bs, 1H, NH), 7.30–7.40 (m, 5 H, ArH).

1-Benzylsulfonyl-2-phenyl-2-pyrroline (2). Colorless crystals, 0.12 g in 1-mmol scale (29%), mp 162°C, $R_f = 0.4$ (PE:EA = 1/1, v/v). IR: 1610 (C=C), 1323 (S=O), 1124 (S=O). ^{1}H NMR (600 MHz) δ: 2.41 (ddd, $J = 18.7$, 14.3, 9.1 Hz, 1H, CH_2), 2.71 (dd, $J =$ 14.3, 7.3 Hz, 1H, CH_2), 4.12 (s, 2H, CH_2), 4.09–4.15 (m, 1H in CH_2), 4.29 (ddd, $J = 16.7$, 8.9, 1.3 Hz, 1H, CH_2), 4.72 (d, $J = 9.9$ Hz, 1H, CH), 7.35–7.37 (m, 2H, ArH), 7.39–7.42 (m, 5H, ArH), 7.45 (t, $J = 7.3$ Hz, 1H, ArH), 7.80 (d, $J = 7.3$ Hz, 2H, ArH). ^{13}C NMR (75 MHz) δ: 27.9, 59.1, 60.4, 68.3, 126.9, 128.4, 128.5, 128.9, 129.1, 130.8, 131.0, 133.0, 165.4. HRMS (ESI) *m/z* $C_{17}H_{19}NO_2S^+$ for $[M + H]^+$: Calcd., 300.1053; Found, 300.1053.

***N*-(2-Hydroxyethyl)benzamide (3a)**. Colorless crystals, 0.17 g (21%); mp 93–95°C,[32] mp 61–63°C. $R_f = 0.50$ (PE: EA = 1:1, v/v). ^{1}H NMR (600 MHz) δ: 3.81 (q, $J = 5.4$ Hz, 2H, CH_2), 3.88 (q, $J = 5.4$ Hz, 2H, CH_2), 4.36 (bs, 1H, OH), 6.67 (bs, 1H, NH), 7.44 (t, $J = 7.2$ Hz, 2H, ArH), 7.52 (t, $J = 7.2$ Hz, 1H, ArH), 7.79 (t, $J = 7.2$ Hz, 2H, ArH). ^{13}C NMR (150 MHz) δ: 41.6, 44.1, 126.9, 128.6, 131.7, 134.1, 167.6.

***N*-(3-Hydroxypropyl)benzamide (3b)**. Colorless crystals, 0.10 g (11%); mp 56–58°C.[33] mp 56–58°C. $R_f = 0.50$ (PE: EA = 1:1, v/v). ^{1}H NMR (600 MHz) δ: 2.09 (q, $J = 6.5$ Hz, 2H, CH_2), 3.58 (t, $J = 6.5$ Hz, 2H, NCH_2), 3.61 (t, $J = 6.5$ Hz, 2H, OCH_2), 6.86 (bs, 1H, NH), 7.39 (t, $J = 7.8$ Hz, 2H, ArH), 7.48 (t, $J = 7.8$ Hz, 1H, ArH), 7.78 (d, $J = 7.8$ Hz, 2H, ArH).

***N*-[3-(2-Formylphenyl)propyl]benzylsulfonamide (4a)**. Colorless oil, 80 mg in 0.5-mmol scale (51%). $R_f = 0.50$ (PE: EA = 1:1, v/v). IR (neat): 3290 (NH), 1693 (C=O), 1322 (S=O), 1151 (S=O), 1083 (S−N). ^{1}H NMR (400 MHz) δ: 10.0 (s, 1H, CHO), 7.71–7.16 (m, 9 H, ArH), 4.19 (s, 2H, CH_2S), 2.97–2.90 (m, 4H, $2CH_2$), 1.68 (q, $J =$ 7.2 Hz, 2H, CH_2). ^{13}C NMR (100 MHz) δ: 193.2, 143.5, 134.3, 133.9, 133. 6, 131.2, 130.6, 129.4, 128.8, 128.7, 126.9, 58.8, 43.2, 32.0, 29.8. HRMS (ESI) *m/z* $C_{17}H_{20}NO_3S^+$ for $[M + H]^+$: Calcd., 318.1158; Found, 318.1160.

***N*-[3-(2-Formylphenyl)propyl]ethanesulfonamide (4b)**. Colorless oil, 90 mg in 0.5-mmol scale (63%). $R_f = 0.50$ (PE:EA = 1/1, v/v). IR (neat): 3286 (NH), 1693 (C=O), 1317 (S=O), 1141 (S=O), 1084 (S−N). ^{1}H NMR (400 MHz) δ: 10.2 (s, 1H, CHO), 7.81–7.30 (m, 4H, ArH), 3.19 (q, $J = 7.2$ Hz, 2H, CH_2), 3.11 (t, $J = 7.2$ Hz, 2H, CH_2), 3.06 (q, $J = 7.6$ Hz, 2H, CH_2), 1.87 (m, 2H, CH_2), 1.38 (t, $J = 7.2$ Hz, 3H, CH_3). ^{13}C NMR (100 MHz) δ: 193.4, 143.6, 134.5, 134.0, 131.3, 126.9, 121.9, 46.8, 42.7, 32.1, 29.9, 8.3. HRMS (ESI) *m/z* $C_{12}H_{18}NO_3S^+$ for $[M + H]^+$: Calcd., 256.1002; Found, 256.1006.

***rel*-(8*S*,9a*S*,12a*S*)-9-Phenyl-5,6,9,9a,14,15-hexahydro-16a*H*-diisoquinolino-[2,1-*b*:2′,1′-*d*][1,2,4]thiadiazine-8,8-dioxide [(±)-*SSS*-5]**. Colorless crystals, 80 mg (4%); mp 145–147°C. $R_f = 0.70$ (PE:EA = 1/2, v/v). IR: 3100 (CH), 1334 (S=O), 1136 (S=O), 1093 (S−N). ^{1}H NMR (400 MHz) δ: 7.33–5.92 (m, 13H, ArH), 5.48 (s, 1H in CH_2), 4.77 (d, $J = 9.6$ Hz, 1H, CH), 4.73 (d, $J = 9.6$ Hz, 1H, CH), 4.34 (m, 1H, CH_2), 4.23 (m, 1H, CH_2), 3.35 (m, 2H, CH_2), 3.31 (m, 2H, CH_2), 3.00 (m, 1H, CH_2), 2.89 (m, 1H, CH_2). ^{13}C NMR (100 MHz) δ: 135.5, 135.2, 134.5, 133.6, 131.9, 129.2, 129.0, 128.7, 128.0, 127.7, 127.4, 126.6, 125.7, 124.8, 79.8, 71.3, 59.3, 47.3, 41.0, 30.0, 28.5. HRMS (ESI) Calcd. $C_{25}H_{25}N_2O_2S^+$ for $[M + H]^+$, 417.1631; Found, 417.1640.

***rel*-(8*R*,9a*R*,12a*S*)-9-Phenyl-5,6,9,9a,14,15-hexahydro-16a*H*-diisoquinolino-[2,1-*b*:2′,1′-*d*][1,2,4]thiadiazine-8,8-dioxide [(±)-*RRS*-5]**. Colorless crystals, 30 mg (2%); mp 126–128°C. $R_f = 0.80$ (PE:EA = 1/2, v/v). IR: 3062 (CH), 1381 (S=O), 1146 (S=O), 919 (S−N). ^{1}H NMR (400 MHz) δ: 7.32–6.17 (m, 13H, ArH), 6.07 (s, 1H, CHN), 5.12 (d, $J = 10.6$ Hz, 1H, CH), 4.58 (d, $J = 10.6$ Hz, 1H, CH), 3.75 (m, 1H, CH_2), 3.62 (m, 1H, CH_2), 3.22 (dt, $J = 14.5$, 10.8 Hz, 1H, CH_2), 3.00 (m, 2H, CH_2), 2.70 (m, 2H, CH_2), 2.67 (m, 1H, CH_2). ^{13}C NMR (100 MHz) δ: 135.6, 134.5, 132.5, 132.0, 129.7, 129.0, 128.9, 128.6, 128.5, 127.9, 127.8, 127.3, 127.0, 124.5, 75.1, 64.8, 63.8, 37.7, 29.2, 29.2, 39.5. HRMS (ESI) *m/z* $C_{25}H_{25}N_2O_2S^+$ for $[M + H]^+$:Calcd., 417.1631; Found, 417.1640.

2-(Benzylsulfonyl)-1,2,3,4-tetrahydroisoquinolin-1-ol (6a). Colorless crystals, 30 mg (2%); mp 204–205°C. $R_f = 0.50$ (PE:EA = 1/2, v/v). IR: 3496 (OH), 3000 (CH), 1333 (S=O), 1146 (S=O), 914 (S−N). ^{1}H NMR (400 MHz) δ: 7.31–7.09 (m, 9H, ArH), 6.02 (d, $J = 5.3$ Hz, 1H, CH), 4.35 (s, 2H, CH_2), 3.55 (ddd, $J = 12.8$, 5.6, 2.4 Hz, 1H, CH_2), 3.30 (dt, $J = 12.4$, 0.4 Hz, 1H, CH_2), 2.94 (d, $J = 5.4$ Hz, 1H, OH), 2.77 (ddd, $J = 16.3$, 11.7, 6.0, Hz, 1H, CH_2), 2.66 (dt, $J = 16.1$, 3.2 Hz, 1H, CH_2). ^{13}C NMR (100 MHz) δ: 134.0, 133.7, 130.9, 130.7, 128.7, 128.6, 128.4, 126.7, 77.1, 59.7, 38.8, 29.0. HRMS (ESI) *m/z* $C_{32}H_{34}N_2NaO_6S_2^+$ for $[2M + Na]^+$: Calcd., 629.1750; Found, 629.1756.

1-Chloro-2-(ethylsulfonyl)-1,2,3,4-tetrahydroisoquinoline (7a). Colorless crystals, 10 mg (2%); mp 170–172°C. $R_f = 0.50$ (PE:EA = 1/1, v/v). IR: 1471 (S=O), 1145 (S=O), 1022 (S−N). ^{1}H NMR (400 MHz) δ: 7.69–7.15 (m, 4H, ArH), 6.74 (s, 1H, CH),

3.89 (ddt, $J = 13.2, 5.6, 1.2$ Hz, 1H, CH_2), 3.56 (dt, $J = 12.8, 3.6$ Hz, 1H, CH_2), 3.07 (q, $J = 7.2$ Hz, 2H, CH_2), 3.16–2.98 (m, 1H, CH_2), 2.77 (ddd, $J = 16.4, 3.6, 1.6$ Hz, 1H, CH_2), 1.26 (t, $J = 7.2$ Hz, 3H, CH_3). ^{13}C NMR (100 MHz) δ: 135.6, 130.1, 129.6, 129.0, 128.3, 126.7, 85.9, 47.4, 38.8, 28.9, 7.7. HRMS (ESI) *m/z* $C_{11}H_{14}NO_2S^+$ for $[M - Cl]^+$: Calcd., 224.0740; Found, 224.0740.

Reaction of Benzylsulfonyl Chloride and *N*-Benzylidenebenzylamine in the Presence of Triethylamine

To a solution of *N*-benzylidenebenzylamine (1.31 g, 6.7 mmol) and triethylamine (0.97 g, 9.63 mmol) in anhydrous THF (10 mL) was added dropwise a solution of benzylsulfonyl chloride (1.4 g, 7.4 mmol) in anhydrous THF (10 mL) in an ice-water bath. After the addition, the resulting mixture was further stirred in the ice-water bath for 24 h and at r.t. for 48 h. After the filtration of salts and removal of solvent, the residue was purified via silica gel column chromatography (PE/EA 50:1, v/v) to afford colorless crystals 1.9 g (20%), mp 132–134°C.[32] mp 125–127°C. $R_f = 0.8$ (PE/EA = 30:1). ^{1}H NMR (300 MHz) δ: 7.12 (s, 2H, 2CH), 7.25–7.54 (m, 10 H, ArH). ^{13}C NMR (75 MHz) δ: 126.5, 127.6, 128.6, 137.3.

Reaction of 4-Nitrobenzenesulfonyl Chloride and 3,4-Dihydroisoquinoline in Toluene with Oxirane as Additive

To a solution of 3,4-dihydroisoquinoline (1.31 g, 10 mmol) and oxirane (0.88 g, 1 mL, 20 mmol) in anhydrous toluene (10 mL) was added dropwise a solution of 4-nitrobenzenesulfonyl chloride (1.11 g, 5 mmol) in anhydrous toluene (10 mL) in an ice-water bath. After addition, the resulting mixture was further stirred in the ice-water bath for 24 h and at r.t. for 48 h. After the filtration of salts and removal of solvent, the residue was purified via silica gel column chromatography (PE/EA 3:1, v/v) to afford colorless crystals.

2-(4-Nitrophenylsulfonyl)-1,2,3,4-tetrahydroisoquinolin-1-ol (6b). Light-brown crystals, 40 mg (3%); mp 136–138°C. $R_f = 0.6$ (PE:EA = 1/3, v/v). IR: 3491 (OH), 1356 (S=O), 1176 (S=O), 943 (S−N). ^{1}H NMR (400 MHz) δ: 8.32(d, $J = 8.8$ Hz, 2H, ArH), 8.10 (d, $J = 8.8$ Hz, 2H, ArH), 7.27–7.12 (m, 4H, ArH), 6.36 (d, $J = 5.6$ Hz, 1H, CH), 3.87 (dddd, $J = 12.8, 5.6, 2.4, 0.8$ Hz, 1H, CH_2), 3.40 (dt, $J = 12.0, 8.4$ Hz, 1H in CH_2), 2.95 (ddd, $J = 16.4, 12.0, 6.0$ Hz, 1H, CH_2), 2.90 (d, $J = 5.6$ Hz, 1H, OH), 2.82 (dt, $J = 19.6, 3.4$ Hz, 1H, CH_2). ^{13}C NMR (100 MHz) δ: 150.1, 146.0, 133.8, 133.2, 129.1, 128.7, 128.3, 127.1, 124.2, 77.2, 38.4, 28.7. HRMS (ESI) *m/z* $C_{15}H_{15}N_2O_5S^+$ for $[M + H]^+$: Calcd., 335.0696; Found, 335.0704.

1-(2-Chloroethoxy)-2-(4-nitrophenylsulfonyl)-1,2,3,4-tetrahydroisoquinoline (8a). Colorless oil, 0.12 g (7%); $R_f = 0.5$ (PE:EA = 1/3, v/v). IR: 3104 (CH), 1383 (S=O), 1167 (S=O), 945 (S−N). ^{1}H NMR (400 MHz) δ: 8.23 (d, $J = 8.8$ Hz, 2H, ArH), 7.93 (d, $J = 8.8$ Hz, 2H, ArH), 7.27–6.98 (m, 4H, ArH), 6.11 (s, 1H, CH), 4.01 (ddd, $J = 11.2, 6.0, 0.8$ Hz, 1H, CH_2), 3.93 (ddd, $J = 11.2, 6.0, 4.4$ Hz, 1H, CH_2), 3.82 (dddd, $J = 14.0, 6.0, 3.2, 0.8$ Hz, 1H, CH_2), 3.71–3.62 (m, 3H, 1H, CH_2, CH_2), 2.69 (dt, $J = 4.0, 16.4$ Hz, 1H, CH_2), 2.49 (ddd, $J = 16.4, 11.2, 6.0$ Hz, 1H in CH_2). ^{13}C NMR (100 MHz) δ: 149.9, 146.3, 133.1, 132.1, 129.2, 128.7, 128.5, 128.1, 126.9, 124.2, 83.6, 67.8, 42.8, 39.0, 26.6. HRMS (ESI) *m/z* $C_{17}H_{17}ClN_2O_5NaS^+$ for $[M + Na]^+$: Calcd., 419.0439; Found, 419.0433.

Reaction of 4-Nitrobenzenesulfonyl Chloride with 3,4-Dihydroisoquinoline in 1,4-Dioxane

To a solution of 3,4-dihydroisoquinoline (1.31 g, 10 mmol) in anhydrous 1,4-dioxane (10 mL) was added dropwise a solution of 4-nitrobenzenesulfonyl chloride (1.11 g, 5 mmol) in anhydrous 1,4-dioxane (10 mL) in an ice-water bath. After addition, the resulting mixture was further stirred in the ice-water bath for 24 h and at r.t. for 48 h. After the filtration of salts and removal of solvent, the residue was purified via silica gel column chromatography (PE/EA 50:1, v/v) to afford colorless crystals.

1-Chloro-2-(4-nitrophenylsulfonyl)-1,2,3,4-tetrahydroisoquinoline (7b). Colorless crystals, 80 mg (5%); mp 215°C. R_f = 0.5 (PE:EA = 1/3, v/v). IR: 3100 (CH), 1347 (S=O), 1168 (S=O), 963 (S−N). ^{1}H NMR (400 MHz) δ: 8.26 (d, J = 9.2 Hz, 2H, ArH), 8.10 (d, J = 9.2 Hz, 2H, ArH), 7.19–6.93 (m, 4H, ArH), 6.81 (s, 1H, CH), 4.08–4.02 (m, 1H, CH_2), 3.67 (ddd, J = 13.6, 11.2, 5.6 Hz, 1H, CH_2), 2.66–2.51 (m, 2H, CH_2). ^{13}C NMR (100 MHz) δ: 150.1, 146.4, 132.8, 132.1, 128.9, 128.6, 128.4, 128.2, 127.1, 124.5, 81.9, 39.5, 26.8. HRMS (ESI) *m/z* $C_{15}H_{13}N_2O_4S^+$ for $[M - Cl]^+$: Calcd., 317.0591; Found, 317.0594.

1-Ethoxy-2-(4-nitrophenylsulfonyl)-1,2,3,4-tetrahydroisoquinoline (9). Light-yellow granular solid, 0.11 g (6%); mp 78–80°C. R_f = 0.7 (PE:EA = 1/3, v/v). IR: 3099 (CH), 1351 (S=O), 1168 (S=O), 1110 (C−O−C), 918 (S−N). ^{1}H NMR (400 MHz) δ: 8.22(d, J = 8.8 Hz, 2H, ArH), 7.93 (d, J = 8.8 Hz, 2H, ArH), 7.23–6.97 (m, 4H, ArH), 6.04 (s, 1H, CH), 3.85–3.74 (m, 1H, CH_2), 3.68–3.61 (m, 1H, CH_2), 3.76–3.66 (q, J = 6.8 Hz, 2H, CH_2), 2.68 (ddd, J = 16.8, 4.4, 3.2 Hz, 1H, CH_2), 2.50 (ddd, J = 16.4, 11.6, 6.4 Hz, 1H, CH_2), 1.12 (t, J = 6.8 Hz, 3H, CH_3). ^{13}C NMR (100 MHz) δ: 149.9, 146.6, 133.1, 132.8, 128.9, 128.7, 128.3, 128.1, 126.8, 124.1, 83.4, 63.6, 38.9, 26.8, 14.9. HRMS (ESI) *m/z* $C_{17}H_{18}N_2O_5NaS^+$ $[M + Na]^+$: Calcd., 385.0829; Found, 385.0828.

CCDC-831814 contains the supplementary crystallographic data of **9**. These data can be obtained free of charge via www.ccdc.cam.ac.uk/retrieving.html or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge, B2 1EZ, UK (fax +44-1223-336033; E-mail: deposit@ccdc.cam.ac.uk).

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