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IODOTRIMETHYLSILANE AND CATALYTIC IODINE PROMOTED CYCLIZATION FOR THE FACILE SYNTHESIS OF 3-MONOARYLATED FIVE-MEMBERED BENZOSULTAMS

Yue-Hui Dong,[1,2] Qiong-Wei Ni,[1] Shu-Tao Ma,[1] and Zhao-Peng Liu[1]*

[1]Department of Organic Chemistry, School of Pharmaceutical Sciences, Shandong University, Jinan 250012, P.R. China. E-mail: liuzhaop@sdu.edu.cn
[2]Jinan Health School of Shandong Province, Jinan 250023, China

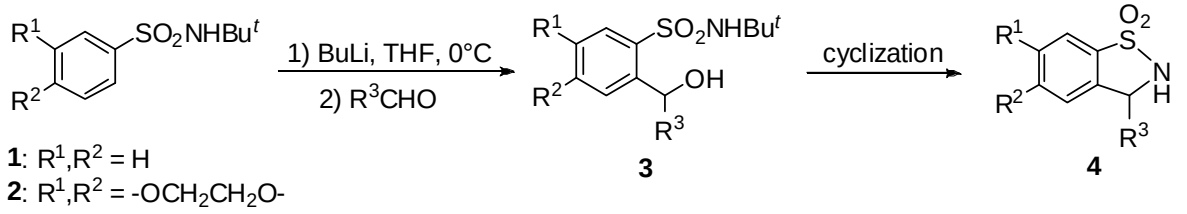
Abstract – 3-Monoarylated five-membered benzosultams with various functional groups were prepared by simple and convenient two-step procedures. *N*-*t*-Bu-benzenesulfonamides were lithiated and reacted with substituted aromatic aldehydes to form the corresponding carbinols, which were converted to the cyclic compounds via a sequence of consecutive processes mediated by TMSCl-NaI-MeCN reagent and catalytic amount of iodine. Iodine played a crucial role in the eliminating the reductive side reaction in the cyclization processes.

1. INTRODUCTION

The sulfonamide functional group stands out as one of the most important pharmacophores and is widely used by medicinal chemists for the design of a host of biologically active derivatives with pharmacological applications.[1] Recently, high interest has also been directed to their conformationally constrained cyclic counterparts, the sultams, which display a vast array of biological activities, act as nonsteroidal antiinflammatory agents, agonists of 5-HT_{1A} receptors, novel serine inhibitors, zinc enzyme carbonic anhydrase inhibitors, etc.[2] 3-substituted five-membered benzosultams have also received attention as potent modulators of neurotransmitters,[3] HIV-1 inhibitors,[4] and serotonin antagonists.[5] In addition to their significance in the treatment of diseases, sultams have also been succesfully applied as chiral auxiliaries in asymmetric versions of several reactions, including alkylations, acylations, aldol reactions, Diels-Alder reactions and azidations.[6] Design and synthesis of *N*-fluorobenzosultam based on sultam templates have also become one of the important strategies for the development of novel electrophilic fluorinating agents.[7]

As part of a program to study the biological activities of highly functionalized cyclic sulfonamides, we

are interested in the development of an efficient method for the construction of 3-monoarylated five-membered benzosultams. An often used method for the preparation of such kind of compounds includes two steps from saccharin: (1) the direct nucleophilic addition to the carbonyl carbon using strong nucleophiles such as aryllithium reagents or Grignard reagents to form a cyclic *N*-sulfonylimine; (2) reduction of the sulfonylimine through Pd/C catalyzed hydrogenation.[8] But this method has limitations owning to the unavailability of some of the functionalized organometallic species, or the poor reactivities of some hindered organometallic reagents with saccharin. In addition, substituted saccharins on the aromatic ring are not readily available and usually take several steps to prepare.[9] In fact, only a limited number of 3-monoarylated five-membered benzosultams have been prepared in this way. In recent years, metal (Fe, Mn, Ru, Cu, Rh, Co) complexes catalyzed intramolecular C–H amination and intramolecular aziridination have been investigated for the synthesis of five- and six-membered benzosultams.[10] However, a simple and general procedure to generate 3-monoarylated five-membered benzosultams, which can tolerate a wide variety of functional groups, remains elusive. In previous researches, we developed a novel cyclization method mediated by iodotrimethylsilane (Me_3SiI, TMSI), generated *in situ* by mixing chlorotrimethylsilane (TMSCl) with sodium iodide (NaI) in acetonitrile (MeCN) solution, for the efficient construction of 3,3-disubstituted five-membered benzosultams, 3-monosubstituted and 3,3-disubstituted six-membered benzosultams.[11] To study the mechanism, the scope and limitations of this novel methodology, we want to adopt the same two-step strategy for the preparation of 3-monoarylated five-membered benzosultams (Scheme 1). We here report our new findings in the synthesis of 3-monoarylated benzosultams.



Scheme 1. Two-step Synthesis of 3-Monoarylated Five-membered Benzosultams

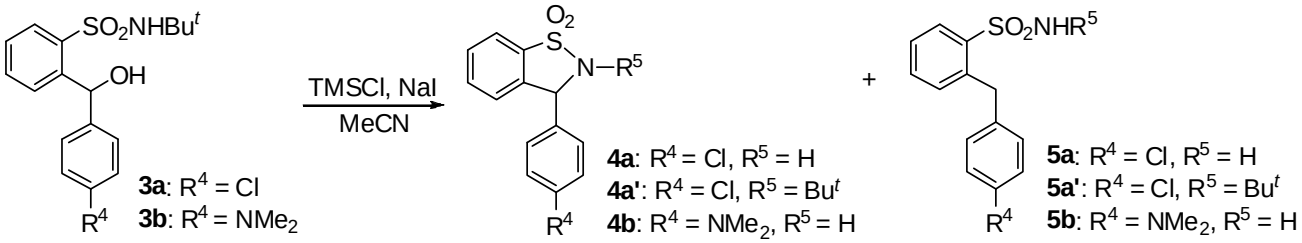
2. RESULTS AND DISCUSSION

The first step takes advantage of the powerful sulfonamide directed *ortho* metalation (D*o*M) effect.[12] *N*-*t*-Butylbenzenesulfonamides **1** or **2** underwent *ortho* metalation, as well as *N*-metalation, with two equivalents of BuLi in anhydrous THF at 0 °C for 30 min under a nitrogen atmosphere to generate dilithiosulfonamide, which was reacted with an aldehyde to form a carbinol. Keen to test the generality of our strategy, a variety of aromatic aldehydes with different functional groups were applied, and the corresponding carbinol sulfonamides **3a-m** were obtained in general good yields, ranging from 58 to 96%

(Table 1). In next step, we first tested the TMSCl-NaI-MeCN reagent system to effect the cyclization. When carbinol sulfonamides **3a** and **3b** were treated with two equivalents of TMSCl-NaI in acetonitrile under reflux conditions for one hour respectively, to our surprise, in addition to the normal cyclization products **4a** and **4b**, there were also the unexpected deoxygenated products **5a** and **5b** formed (Scheme 2), and the ratio of **4a** to **5a** is 1:2, while **4b** to **5b** is 5:1! (Table 2, entries 1 and 2). When we run the same reaction with substrate **3a** at room temperature, the cyclic *N*-*t*-butylbenzosultam **4a'**, together with the deoxygenated *N*-*t*-butylbenzenesulfonamide **5a'** were isolated, the ratio of **4a'** to **5a'** is 1:2 (Table 2, entriy 5). It is clear now, in the TMSCl-NaI-MeCN reagent promoted synthesis of five-membered benzosultams, the cyclization goes first, while the removal of the *t*-butyl protective group runs second, and the later needs heating to go to completion. It is also apparent that the reductive reaction is competitive with the cyclization, but how is the reduction product generated?

Table 1. Synthesis of Carbinol **3a-m** and Benzosultams **4a-m**

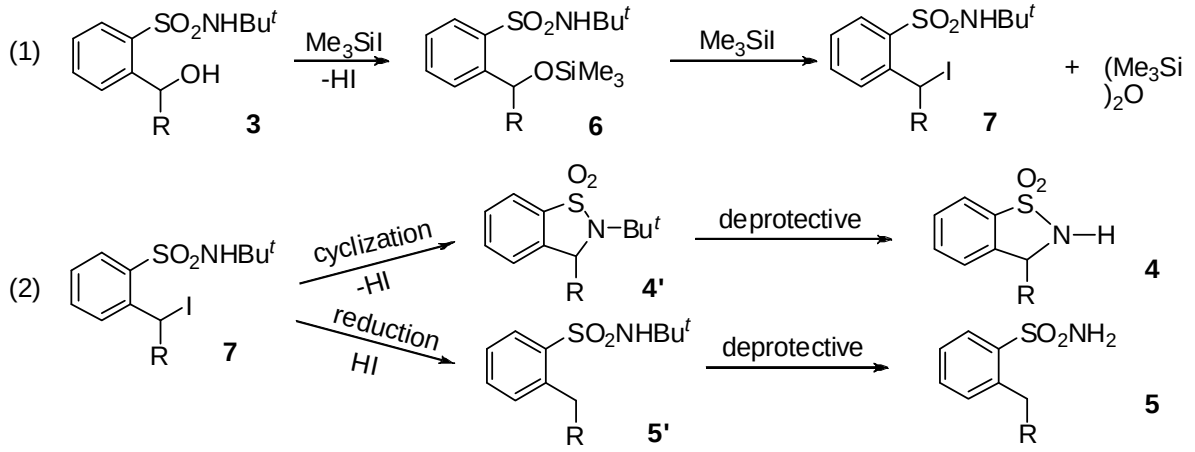
Entry	R^1	R^2	R^3	Yields (%)	
				3a-m	**4a-m**
a	H	H	4-chlorophenyl	91	90
b	H	H	4-dimethylaminophenyl	58	82
c	H	H	4-methylphenyl	86	89
d	H	H	4-methoxyphenyl	90	86
e	H	H	3,4-dimethoxyphenyl	86	84
f	H	H	3,4,5-trimethoxyphenyl	96	75
g	$-OCH_2O-$		4-chlorophenyl	76	81
h	$-OCH_2O-$		4-methoxyphenyl	88	91
i	$-OCH_2O-$		3,4,5-trimethoxyphenyl	98	76
j	$-OCH_2O-$		3,4-dimethoxyphenyl	80	84
k	$-OCH_2O-$		4-methylphenyl	88	83
l	$-OCH_2O-$		4-fluorophenyl	90	80
m	$-OCH_2O-$		3-chlorophenyl	80	76



Scheme 2. TMSCl-NaI-MeCN Reagent Mediated Cyclization of Carbinol Sulfonamides **3a** and **3b**

It is well known that alcohols can be converted to iodides by the iodotrimethylsilane rather rapidly.[13] Therefore the novel cyclization mediated by iodotrimethylsilane can be visulised to proceed via a sequence of consecutive processes, involving in the conversion of the hydroxy group to iodide, an intramolecular necleophilic substitution for the cyclization, and finally the removal of the *t*-butyl

protective group (Scheme 3). In the formation of an iodide, an alcohol first reacts with one molecule of iodotrimethylsilane to form trimethylsilyl ether **6**, and in this process, one molecule of HI is always generated.[14] We postulate that the hydrogen iodide might account for the formation of the deoxygenated product, as HI is known to reduce an alkyl iodide to an alkane. Comparing with **4a** to **5a**, the reversed ratio of **4b** to **5b** also supports our hypothesis, since the dimethylamine group in the substrate **3b** may trap part of the HI, so the deoxygenated product **5b** was much less than **5a**, while the cyclization product **4b** was predominant.



Scheme 3. Mechanism Considerations of Iodotrimethylsilane Promoted Cyclization Processes

Table 2. The Results of Cyclization Under Different Synthetic Conditions

Entry	Substrate	TMSCl-NaI	Additive	Temperature	Product and Yields (%)
1	**3a**	2 eq		reflux	**4a**, 32% **5a,** 67%
2	**3b**	2 eq		reflux	**5a**, 80% **5a,** 17%
3	**3a**	2 eq	Et_3N, 1 eq	reflux	**4a'**, 21%
4	**3a**	3 eq	Et_3N, 1 eq	reflux	**4a**, 79% **5a,** 10%
5	**3a**	2 eq		rt	**4a'**, 29% **5a',** 60%
6	**3a**	2 eq	I_2, 0.5 eq	reflux	**4a**, 90%

To prove our above assumptions, we first used triethylamine as HI scavenger. When carbinol sulfonamides **3a** was reacted with two equiv of TMSCl and NaI in acetonitrile and one equiv of triethylamine under reflux conditions (Table 2, entry 3), indeed, there were no deoxygenated products **5a** or **5a'** detected, however, the reaction did not go completely, with 76% of **3a** recovered, and the cyclic product was identified by ^{1}H NMR analysis as *N*-*t*-butylbenzosultam **4a'**. It is clear that HI do play an important role in the formation of the deoxygenated products and triethylamine is an effective HI scavenger. But triethylamine can also interact directly with iodotrimethylsilane, reducing its lewis acidity and nucleophilicity,[15] thus affect the formation of the iodide **7** and the cyclization process. When one more equiv of TMSCl-NaI were added to above reaction mixture, the transformation was completed in

less than one hour, but in addition to the normal cyclic product **4a**, there were still about 10% deoxygenated products **5a** produced (Table 2, entry 4). Other HI scavengers like *N*,*N*-dimethylaniline, cyclohexene and sodium carbonate did not give any better results than triethylamine. Inspired by the report that iodine exerts a catalytic effect in the conversion of aromatic esters with iodotrimethylsilane,[16] we tested iodine as a catalyst for this reaction. When 0.5 equiv of iodine was coexistence in the iodotrimethylsilane reagent system, the cyclization went smoothly with no reduced products **5a** or **5a'** detected by TLC analysis, and the sultam **4a** was obtained in 90% yield after silica gel chromatography (Table 2, entry 6). Though the exact role of iodine needs further experiments to elucidate, it is suggested that iodine sets up an equilibrium with TMSI to form trimethylsilyl triiodide ($TMSI_3$).[16] The triiodide-silicon bond would be expected to be more polarizable than the silicon-iodine bond in iodotrimethylsilane, and we suppose it would behave differently with TMSI, possibly via an alternative pathway involving a six-centered transition state in the transformation of alcohol into the iodide, with the release of Me_3SiOH and I_2, but accompanying no HI formation in this process, thus avoids the formation of the reduction products. There is another possibility that iodine might trap HI through the direct formation of HI-I_2 complex or hydrogen triiodide,[17] affecting the reductive ability of hydrogen iodide.

The scope of this novel cyclization was studied with various carbinol sulfonamides. When **3b-m** were subject to two equiv of TMSCl-NaI and 0.5 equiv of iodine in acetonitrile under reflux conditions for 1 h, sultams **4b-m** were obtained in high yields (Table 1). It was seen that the novel process mediated by TMSI in the presence of catalytic amounts of iodine was effective and tolerant to a broad range of functional groups. It is also of note that the five-membered polymethoxyphenolic benzosultams, the potentially biologically interesting molecules that have the structural features to combine the important sulfonamide pharmacophore and the common polymethoxyphenolic units existed in many bioactive natural products, are readily prepared by this novel method.

In conclusion, we have developed a simple two-step synthesis of 3-monoarylated five-membered benzosultams via the novel cyclization mediated by iodotrimethylsilane and a catalytic amount of iodine. This method makes 3-monoarylated five-membered benzosultams having diverse functional groups easily preparable in two steps.

EXPERIMENTAL

Melting points were determined on an X-6 micro-melting point apparatus (Beijing Tech. Co., Ltd) and were uncorrected. IR spectra (cm^{-1}) were recorded on a Perkin-Elmer 1600 spectrometer. 1H NMR (600 MHz) spectra were recorded at room temperature for $CDCl_3$. All chemical shifts were reported as δ values (ppm) relative to Me_4Si (0.00 ppm) as internal standards for ^1H spectra. Electrospray-ionization mass spectrometry (ESI-MS) was performed on an API 4000 instrument. Microanalyses were performed with a YANAKO CHN-coder MT-5. Column chromatography was performed on silica gel (200-300

mesh). All reactions involving oxygen- or moisture-sensitive compounds were carried out under a dry N_2 atmosphere. Unless otherwise noted, reagents were added by syringe. THF was distilled from sodium/benzophenone immediately prior to use.

Typical Procedure for the Preparation of Carbinol Sulfonamide 3a-m. A 2.5 M solution of BuLi (6.5 mL, 16 mmol) in hexane was added dropwise to a stirred solution of *N*-*t*-Bu-benzenesulfonamide **1** (1.70 g, 8 mmol) in THF (8 mL) under nitrogen at 0 °C. After stirring for 30 min, another solution of 4-chlorobenzaldehyde (1.12 g, 8 mmol) in THF (5 mL) was added. The mixture was stirred for 1 h and quenched by saturated aqueous NH_4Cl. The mixture was then extracted with EtOAc, and the combined organic layers were dried (Na_2SO_4), concentrated in vacuo. Crystallization from EtOAc/petroleum ether gave **3a** as a white solid (2.56 g, 91%), mp 109–111 °C; IR (KBr) 3484, 3270, 1489, 1393, 1300, 1152, 1012, 861, 762 cm^{-1}; ^1H NMR δ 8.05 (dd, *J* = 7.9, 1.2 Hz, 1H), 7.46 (td, *J* = 7.9, 1.2 Hz, 1H), 7.38 (td, *J* = 7.7, 1.0 Hz, 1H), 7.31–7.32 (m, 4H), 7.22 (dd, *J* = 7.7, 1.0 Hz, 1H), 6.71 (s, 1H), 4.91 (s, 1H), 3.51 (s 1H), 1.20 (s, 9H) ; MS (ESI) *m*/*z* 376.4 $[M+Na]^+$. Anal. Calcd for $C_{17}H_{20}ClNO_3S$: C, 57.70; H, 5.70; N, 3.96. Found: C, 57.79; H, 5.81; N, 3.91.

***N*-*t*-Butyl-2-[1-(4-dimethylaminophenyl)-1-hydroxy]methylbenzenesulfonamide 3b.** White solid; mp 117–118 °C; IR (KBr) 3494, 3272, 1613, 1518, 1442, 1302, 1153, 770 cm^{-1}; ^1H NMR δ 8.08 (dd, *J* = 8.3, 1.1 Hz, 1H), 7.54 (td, *J* = 7.5, 1.0 Hz, 1H), 7.41 (td, *J* = 7.7, 1.2 Hz, 1H), 7.26–7.30 (m, 3 H,), 6.75 (d, *J* = 1.5 Hz, 2H), 6.67 (d, *J* = 3.5 Hz, 1H), 4.22 (d, *J* = 3.5 Hz, 1H), 2.97 (s, 6 H), 1.06 (s, 9H); MS *m/z* 363 $[M+H]^+$. Anal. Calcd for $C_{19}H_{26}N_2O_3S$: C, 62.96; H, 7.23; N, 7.73. Found: C, 62.71; H, 7.26; N, 7.68.

***N*-*t*-Butyl-2-[1-(4-methylphenyl)-1-hydroxy]methylbenzenesulfonamide 3c.** White solid; mp 100–101 °C; IR (KBr) 3477, 3273, 1511, 1471,1390, 1152, 1004, 820, 767 cm^{-1}; ^1H NMR δ 8.07 (dd, *J* = 5.9, 0.9 Hz, 1H), 7.38–7.53 (m, 3H), 7.30 (d, *J* = 6.0 Hz, 2H), 7.19 (d, *J* = 5.9 Hz, 2H), 6.68 (s, 1H), 2.35 (s, 3H), 1.11 (s, 9H); MS *m/z* 356.5 $[M+Na]^+$. Anal. Calcd for $C_{18}H_{23}NO_3S$: C, 64.84; H, 6.95; N, 4.20. Found: C, 64.75; H, 6.99; N, 4.15.

***N*-*t*-Butyl-2-[1-(4-methoxylphenyl)-1-hydroxy]methylbenzenesulfonamide 3d.** White solid; mp 103–104 °C; IR (KBr) 3424, 3237, 1614, 1512, 1320, 1245, 1150, 1036, 841, 764 cm^{-1}; ^1H NMR δ 7.74 (dd, *J* = 4.7, 1.6 Hz, 1H), 7.42–7.46 (m, 2H), 7.33–7.37 (m, 2H), 7.02–7.04 (m, 1H), 6.84–6.87 (m, 2H), 5.60 (s, 1H), 3.78 (s, 3H), 1.45 (s, 9H); MS *m/z* 372.3 $[M+Na]^+$. Anal. Calcd for $C_{18}H_{23}NO_4S$: C, 61.87; H, 6.63; N, 4.01. Found: C, 61.69; H, 6.53; N, 4.05.

***N*-*t*-Butyl-2-[1-(3,4-dimethoxylphenyl)-1-hydroxy]methylbenzenesulfonamide 3e.** White solid; mp 133–135 °C; IR (KBr) 3494, 3270, 1606, 1511, 1305, 1265, 1144, 1120, 1037, 812, 757 cm^{-1}; ^1H NMR δ 7.75 (dd, *J* = 4.4, 1.4 Hz, 1H), 7.39–7.51 (m, 3H), 7.06–7.08 (m, 1H), 6.98 (d, *J* = 1.5 Hz, 1H), 6.82 (d, *J* = 6.0 Hz, 1H), 5.58 (s, 1H), 3.86 (s, 3H), 3.82 (s, 3H), 1.46 (s, 9H); MS *m/z* 402.5 $[M+Na]^+$. Anal. Calcd for $C_{19}H_{25}NO_5S$: C, 60.14; H, 6.64; N, 3.69. Found: C, 60.01; H, 6.57; N, 3.65.

***N*-*t*-Butyl-2-[1-(3,4,5-triimethoxylphenyl)-1-hydroxy]methylbenzenesulfonamide 3f.** White solid; mp

122–124 °C; IR (KBr) 3498, 3218, 1592, 1505, 1332, 1230, 1144, 1122, 863, 810, 771 cm^{-1}; ^1H NMR δ 8.08 (dd, *J* = 7.9, 0.9 Hz, 1H), 7.52 (td, *J* = 7.5, 1.0 Hz, 1H), 7.40–7.43 (m, 2H), 6.68 (s, 2H), 6.66 (s, 1H), 3.85 (s, 3H), 3.83 (s, 6H), 1.17 (s, 9H); MS *m/z* 427.5 $[M+NH_4]^+$. Anal. Calcd for $C_{20}H_{27}NO_6S$: C, 58.66; H, 6.65; N, 3.42. Found: C, 58.45; H, 6.78; N, 3.36.

***N*-*t*-Butyl-6-[1-(4-chlorophenyl)-1-hydroxy]methylbenzo[*d*][1,3]dioxole-5-sulfonamide 3g.** White solid; mp 132.5–134.5 °C; IR (KBr) 3524, 3256, 1625, 1597, 1297, 1254, 1139, 814, 768 cm^{-1}; ^1H NMR δ 7.70 (d, *J* = 8.4 Hz, 1H), 7.39 (d, *J* = 8.4 Hz, 2H), 7.34 (dd, *J* = 7.6, 2.0 Hz, 2H), 6.85 (d, *J* = 8.4 Hz, 1H), 6.51 (s, 1H), 6.09 (d, *J* = 1.2 Hz, 1H), 6.01 (d, *J* = 1.2 Hz, 1H), 1.13 (s, 9H); MS *m*/*z* 396.3 $[M-H]^-$. Anal. Calcd for $C_{18}H_{20}ClNO_5S$: C, 54.34; H, 5.07; N, 3.52. Found: C, 54.24; H, 5.15; N, 3.47.

***N*-*t*-Butyl-6-[1-(4-methoxyphenyl)-1-hydroxy]methylbenzo[*d*][1,3]dioxole-5-sulfonamide 3h.** White solid; mp 84–86 °C; IR (KBr): 3522, 3264, 1610, 1512, 1297, 1136, 830, 768 cm^{-1}; ^1H NMR δ 7.45 (dd, *J* = 7.7, 2.0 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 1H), 6.92 (d, *J* = 8.1 Hz, 1H), 6.86 (dd, *J* = 10.7, 2.0 Hz, 2H), 6.01 (d, *J* = 1.3 Hz, 1H), 5.92 (d, *J* = 1.3 Hz, 1H), 5.62 (s, 1H), 3.80 (s, 3H), 1.43 (s, 9H); MS *m*/*z* 392.5 $[M-H]^-$. Anal. Calcd for $C_{19}H_{23}NO_6S$: C, 58.00; H, 5.89; N, 3.56. Found: C, 58.14; H, 5.93; N, 3.47.

***N*-*t*-Butyl-6-[1-(3,4,5-trimethoxyphenyl)-1-hydroxy]methylbenzo[*d*][1,3]dioxole-5-sulfonamide 3i.** White solid; mp 96–98 °C; IR (KBr) 3507, 3365, 1594, 1506, 1331, 1309, 1250, 1128, 1060, 901, 759 cm^{-1}; ^1H NMR δ 7.71 (d, *J* = 8.4 Hz, 1H), 6.86 (d, *J* = 8.4 Hz, 1H), 6.70 (s, 2H), 6.45 (s, 1H), 6.16 (d, *J* = 1.4 Hz, 1H), 6.08 (d, *J* = 1.4 Hz, 1H), 3.85 (s, 9H), 1.08 (s, 9H); MS *m*/*z* 452.5 $[M-H]^-$. Anal. Calcd for $C_{21}H_{27}NO_8S$: C, 55.62; H, 6.00; N, 3.09. Found: C, 55.45; H, 6.10; N, 3.02.

***N*-*t*-Butyl-6-[1-(3,4-dimethoxyphenyl)-1-hydroxy]methylbenzo[*d*][1,3]dioxole-5-sulfonamide 3j.** White solid; mp 84.5–86.5 °C; IR (KBr): 3573, 3274, 1593, 1518, 1304, 1258, 1132, 1000, 898, 758 cm^{-1}; ^1H NMR δ 7.30 (d, *J* = 8.1 Hz, 1H), 7.13 (d, *J* = 2.0 Hz, 1H), 7.09 (dd, *J* = 9.3, 2.0 Hz, 1H), 6.92 (d, *J* = 8.1Hz, 1H), 6.84 (s, 1H), 6.82 (d, *J* = 8.1 Hz, 1H), 6.02 (d, *J* = 1.3 Hz, 1H), 5.95 (d, *J* = 1.3 Hz, 1H), 5.62 (s, 1H), 3.88 (s, 3H), 3.86 (s, 3H), 1.46 (s, 9H); MS *m*/*z* 422.4 $[M-H]^-$. Anal. Calcd for $C_{20}H_{25}NO_7S$: C, 56.72; H, 5.95; N, 3.31. Found: C, 56.53; H, 6.03; N, 3.19.

***N*-*t*-Butyl-6-[1-(4-methylphenyl)-1-hydroxy]methylbenzo[*d*][1,3]dioxole-5-sulfonamide 3k.** White solid; mp 123–125 °C; IR (KBr) 3538, 3246, 1597, 1459, 1299, 1252, 1139, 1004, 901, 816, 766 cm^{-1}; ^1H NMR δ 7.68 (d, *J* = 8.4 Hz, 1H) , 7.33 (d, *J* = 7.9 Hz, 2H), 7.19 (d, *J* = 7.9 Hz, 2H), 6.84 (d, *J* = 8.4 Hz, 1H), 6.48 (d, *J* = 10.4 Hz, 1H), 6.13 (d, *J* = 1.2 Hz, 1H), 6.07 (d, *J* = 1.2 Hz, 1H), 4.45 (d, *J* = 10.7 Hz, 1H), 3.53 (s, 1H), 2.35 (s, 3H), 1.02 (s, 9H); MS *m*/*z* 376.6 $[M-H]^-$. Anal. Calcd for $C_{19}H_{23}NO_5S$: C, 60.46; H, 6.14; N, 3.71. Found: C, 60.31; H, 6.25; N, 3.65.

***N*-*t*-Butyl-6-[1-(4-fluorophenyl)-1-hydroxy]methylbenzo[*d*][1,3]dioxole-5-sulfonamide 3l.** White solid; mp 127–129 °C; IR (KBr) 3439, 3260, 1603, 1509, 1459, 1297, 1253, 1137, 1057, 935, 838, 764 cm^{-1}; ^1H NMR δ 7.70 (d, *J* = 8.4 Hz, 1H), 7.41 (dd, *J* = 8.4, 5.5 Hz, 2H), 7.05 (td, *J* = 7.7, 2.0 Hz, 2H), 6.85 (d, *J* = 8.4 Hz, 1H), 6.52 (s, 1H), 6.09 (d, *J* = 1.2 Hz, 1H), 6.01 (d, *J* = 1.2 Hz, 1H), 3.90 (s, 1H), 1.12

(s, 9H); MS *m*/*z* 380.6 $[M–H]^–$. Anal. Calcd for $C_{18}H_{20}FNO_5S$: C, 56.68; H, 5.29; N, 3.67. Found: C, 56.56; H, 5.33; N, 3.53.

***N*-*t*-Butyl-6-[1-(3-chlorophenyl)-1-hydroxy]methylbenzo[*d*][1,3]dioxole-5-sulfonamide 3m.** White solid; mp 130–132 °C; IR (KBr) 3528, 3317, 1596, 1462, 1300, 1254, 1177, 1135, 991, 900, 818, 767 $cm^{–1}$; ^1H NMR δ 7.70 (d, *J* = 8.4 Hz, 1H), 7.38 (d, *J* = 7.9 Hz, 2H), 7.31 (t, *J* = 7.9 Hz, 1H), 7.27 (s, 1H), 6.86 (d, *J* = 8.4 Hz, 1H), 6.51 (d, *J* = 9.5 Hz, 1H), 6.11 (d, *J* = 1.2 Hz, 1H), 6.02 (d, *J* = 1.2 Hz, 1H), 4.13 (d, *J* = 9.5 Hz, 1H), 3.84 (s, 1H), 1.12 (s, 9H); MS *m*/*z* 396.3 $[M–H]^–$. Anal. Calcd for $C_{18}H_{20}ClNO_5S$: C, 54.34; H, 5.07; N, 3.52. Found: C, 54.29; H, 5.09; N, 3.45.

TMSCl-NaI-MeCN Reagent Promoted Cyclization of 3a and 3b under Reflux Conditions. Chlorotrimethylsilane (0.26 mL, 2 mmol) was added dropwise to a stirred solution of **3a** (354 mg, 1 mmol) and sodium iodide (300 mg, 2 mmol) in MeCN (10 mL) under nitrogen at room temperature. The mixture was heated under reflux for 1 h, after which it was cooled and 10% sodium thiosulfate solution was added. The mixture was extracted with EtOAc, and the combined organic layers were washed with brine, dried (Na_2SO_4), concentrated *in vacuo*. The residue was purified by preparative TLC (Hex-EtOAc, 5:1) to give **4a** (90 mg, 32%) and **5a** (190 mg, 67%).

In the same way, from **3b** (880 mg, 2.43 mmol), chlorotrimethylsilane (0.63 mL, 4.86 mmol) and sodium iodide (730 mg, 4.86 mmol) in MeCN (20 mL), **4b** (560 mg, 80%) and **5b** (120 mg, 17%) were isolated by silica gel chromatography (CH_2Cl_2-Et_2O, 40:1).

3-(4-Chlorophenyl)-2,3-dihydro-1,2-benzisothiazole 1,1-dioxide 4a. White solid; m.p. 181–182 °C; IR (KBr) 3436, 3281, 1487, 1391, 1290, 1169, 839, 758 $cm^{–1}$; ^1H NMR δ 7.85 (dd, *J* = 5.8, 1.5 Hz, 1H), 7.56–7.59 (m, 2H), 7.37 (dd, *J* = 6.9, 1.1 Hz, 2H), 7.32 (d, *J* = 8.5 Hz, 2H), 7.13 (d, *J* = 6.5 Hz, 1H), 5.71 (d, *J* = 3.6 Hz, 1H), 4.95 (d, *J* = 3.6 Hz, 1H); MS *m*/*z* 280.3 $[M+H]^+$. Anal. calcd for $C_{13}H_{10}ClNO_2S$: C, 55.82; H, 3.60; N, 5.01. Found: C, 55.80; H, 3.58; N, 4.99.

2-(4-Chlorobenzyl)benzenesulfonamide 5a. White solid; mp 137–138 °C; IR (KBr) 3346, 3249, 1489, 1151, 899, 755 $cm^{–1}$; ^1H NMR δ 8.09 (dd, *J* = 5.9, 1.0 Hz, 1H), 7.53 (td, *J* = 5.7, 1.0 Hz, 1H), 7.40 (td, *J* = 5.6, 0.9 Hz, 1H), 7.27–7.30 (m, 3H), 7.15 (d, *J* = 6.3 Hz, 2H), 4.31 (s, 2H), 4.46 (s, 2H) ; MS *m*/*z* 282.0 $[M+H]^+$. Anal. calcd for $C_{13}H_{12}ClNO_2S$: C, 55.42; H, 4.29; N, 4.97. Found: C, 55.36; H, 4.23; N, 4.89.

3-(4-Dimethylaminophenyl)-2,3-dihydro-1,2-benzisothiazole 1,1-dioxide 4b. White solid; mp 137–139 °C; IR (KBr) 3437, 3259, 1623, 1532, 1450, 1324, 1302, 1278, 1164, 818, 765 $cm^{–1}$; ^1H NMR δ 7.85 (dd, *J* = 7.1, 1.0 Hz, 1 H), 7.53–7.58 (m, 2 H), 7.16–7.20 (m, 3 H), 6.72 (s, 2 H), 5.66 (d, *J* = 3.9 Hz, 1 H), 4.70 (d, *J* = 3.9 Hz, 1 H), 2.98 (s, 6 H); MS *m*/*z* 289 $[M+H]^+$. Anal. Calcd for $C_{15}H_{16}N_2O_2S$: C, 62.48; H, 5.59; N, 9.71. Found: C, 62.21; H, 5.56; N, 9.68.

2-(4-Dimethylaminobenzyl)benzenesulfonamide 5b. White solid; mp 209–210 °C; IR (KBr) 3494, 3269, 1511, 1463, 1389, 1305, 1285, 1143, 978, 812, 758 $cm^{–1}$; ^1H NMR δ 8.06 (dd, *J* = 8.2, 1.5 Hz, 1H), 7.55 (td, *J* = 7.5, 1.2 Hz, 1H), 7.39 (d, *J* = 7.0 Hz, 2H), 7.06 (d, *J* = 8.7 Hz, 2H), 6.69 (d, *J* = 8.7 Hz, 2H),

4.14 (s, 2H), 2.93 (s, 6H); MS *m/z* 289.4 $[M–H]^-$. Anal. Calcd for $C_{15}H_{18}N_2O_2S$: C, 62.04; H, 6.25; N, 9.65. Found: C, 62.00; H, 6.21; N, 9.67.

TMSCl-NaI-MeCN Reagent Promoted Cyclization of 3a at RT. Chlorotrimethylsilane (0.26 mL, 2 mmol) was added dropwise to a stirred solution of **3a** (354 mg, 1 mmol) and sodium iodide (300 mg, 2 mmol) in MeCN (10 mL) under nitrogen at room temperature. The mixture was stirred for 1 h. 10% sodium thiosulfate solution was added. The mixture was extracted with EtOAc, and the combined organic layers were washed with brine, dried (Na_2SO_4), concentrated *in vacuo*. The residue was purified by preparative TLC (Hexane-EtOAc, 5:1) to give **4a'** (100 mg, 29%) and **5a'** (200 mg, 60%).

***N*-*t*-Butyl-3-(4-chlorophenyl)-2,3-dihydro-1,2-benzisothiazole 1,1-dione 4a'.** White solid; mp 135–136 °C; IR (KBr) 3323, 3281, 3251, 1487, 1300, 1291, 1169, 839, 758 cm^{-1}; ^1H NMR δ 8.10 (dd, *J* = 4.0, 0.7 Hz, 1H), 7.50 (td, *J* = 3.8, 0.7 Hz, 1H), 7.43 (td, *J* = 3.8, 0.7 Hz, 1H), 7.38 (s, 4H), 7.27–7.29 (m, 1H), 6.73 (s, 1H), 1.23 (s, 9H); MS *m/z* 358.3 $[M+Na]^+$. Anal. Calcd for $C_{17}H_{18}ClNO_2S$: C, 60.80; H, 5.40; N, 4.17. Found: C, 60.73; H, 5.43; N, 4.14.

***N*-*t*-Butyl-2-(4-chlorobenzyl)benzensulfonamide 5a'.** White solid; mp 102–103 °C; IR (KBr) 3301, 1473, 1441, 1307, 1150, 796, 760 cm^{-1}; ^1H NMR δ 8.08 (dd, *J* = 6.0, 0.9 Hz, 1H), 7.47 (td, *J* = 5.7, 1.0 Hz, 1H), 7.36 (td, *J* = 8.0, 0.8 Hz, 1H), 7.27–7.30 (m, 2H), 7.19 (d, *J* = 5.7 Hz, 1H), 7.14 (d, *J* = 6.3 Hz, 2H), 4.43 (s, 2H), 3.97 (s, 1H), 1.08 (s, 9H); MS *m/z* 360.4 $[M+Na]^+$. Anal. Calcd for $C_{17}H_{20}ClNO_2S$: C, 60.43; H, 5.97; N, 4.15. Found: C, 60.46; H, 5.99; N, 4.09..

TMSCl-NaI-MeCN Reagent Promoted Cyclization of 3a in the Presence of Et_3N under Reflux Conditions. Chlorotrimethylsilane (0.65 mL, 5 mmol) was added dropwise to a stirred solution of **3a** (900 mg, 2.55 mmol), sodium iodide (765 mg, 5.10 mmol) in MeCN (20 mL) and triethylamine (0.36 mL, 2.55 mmol) under nitrogen at room temperature. The mixture was heated under reflux for 1 h, after which it was cooled and 10% sodium thiosulfate solution was added. The mixture was extracted with EtOAc, and the combined organic layers were washed with brine, dried (Na_2SO_4), concentrated *in vacuo*. The residue was purified by silica gel chromatography (CH_2Cl_2-Et_2O, 40:1) to give **4a'** (174 mg, 21%) and recovered **3a** (684 mg, 76%).

Typical Procedure for the Preparation of 3-Monoarylated Benzosultam 4 Promoted by Iodotrimethylsilane and Catalytic Amount of Iodine. Chlorotrimethylsilane (0.26 mL, 2 mmol) was added dropwise to a stirred solution of **3a** (0.36 g, 1 mmol), iodine (0.12 g, 0.5 mmol) and sodium iodide (0.30 g, 2 mmol) in MeCN (10 mL) under nitrogen at room temperature. The mixture was heated under reflux for 1 h, after which it was cooled and 10% sodium thiosulfate solution was added. The mixture was extracted with EtOAc, and the combined organic layers were washed with brine, dried (Na_2SO_4), concentrated *in vacuo*. Recrystallizion from EtOAc/hexane gave **4a** as a white solid (0.25 g, 90%).

3-(4-Methylphenyl)-2,3-dihydro-1,2-benzisothiazole 1,1-dioxide 4c. White solid; mp 166–168 °C; IR (KBr): 3277, 3036, 1511, 1454, 1391, 1279, 1168, 1048, 833, 751 cm^{-1}; ^1H NMR δ 7.84 (td, *J* = 2.7, 1.1

Hz, 1H), 7.55 (td, *J* = 2.7, 1.1 Hz, 2H), 7.18–7.25 (m, 4H), 7.12–7.14 (m, 1H), 5.68 (d, *J* = 2.9 Hz, 1H), 4.77 (d, *J* = 2.9 Hz, 1H), 2.36 (s, 3H); MS *m/z* 260.3 $[M+H]^+$. Anal. Calcd for $C_{14}H_{13}NO_2S$: C, 64.84; H, 5.05; N, 5.40. Found: C, 64.67; H, 5.11; N, 5.36.

3-(4-Methoxyphenyl)-2,3-dihydro-1,2-benzisothiazole 1,1-dioxide 4d. White solid; mp 143–144 °C; IR (KBr) 3252, 3010, 1615, 1515, 1292, 1276, 1251, 1165, 1028, 919, 838, 763 cm^{-1}; ^1H NMR δ 7.85 (dd, *J* = 4.2, 1.8 Hz, 1H), 7.55–7.57 (m, 2H), 7.25 –7.26 (m, 2H), 7.14 (d, *J* = 6.5 Hz, 1H), 6.90–6.92 (m, 2H), 5.68 (d, *J* = 3.4 Hz, 1H), 4.77 (d, *J* = 3.4 Hz, 1H), 3.81 (s, 3H); MS *m/z* 276.5 $[M+H]^+$. Anal. Calcd for $C_{14}H_{13}NO_3S$: C, 61.07; H, 4.76; N, 5.09. Found: C, 60.89; H, 4.87; N, 4.93.

3-(3,4-Dimethoxyphenyl)-2,3-dihydro-1,2-benzisothiazole 1,1-dioxide 4e. White solid; mp 184–185 °C; IR (KBr) 3276, 3068, 1598, 1519, 1452, 1377, 1279, 1162, 1027, 864, 752 cm^{-1}; ^1H NMR δ 7.86 (dd, *J* = 6.7, 1.7 Hz, 1H), 7.56–7.61 (m, 2H), 7.17–7.19 (m, 1H), 6.96 (dd, *J* = 8.2, 2.1 Hz, 1H), 6.88 (d, *J* = 8.2 Hz, 1H), 6.84 (d, *J* = 2.1 Hz, 1H), 5.69 (d, *J* = 3.9 Hz, 1H), 4.86 (d, *J* = 3.9 Hz, 1H), 3.90 (s, 3H), 3.84 (s, 3H); MS *m/z* 306.4 $[M+H]^+$. Anal. Calcd for $C_{15}H_{15}NO_4S$: C, 59.00; H, 4.95; N, 4.59. Found: C, 58.86; H, 4.97; N, 4.45.

3-(3,4,5-Trimethoxyphenyl)-2,3-dihydro-1,2-benzisothiazole 1,1-dioxide 4f. White solid; mp 192–194 °C; IR (KBr) 3213, 3006, 1597, 1507, 1467, 1342, 1285, 1165, 1062, 995, 856, 782, 756 cm^{-1}; ^1H NMR δ 7.85 (dd, *J* = 4.8, 1.4 Hz, 1H), 7.56–7.61 (m, 2H), 7.20–7.22 (m, 1H), 6.59 (s, 2H), 5.64 (s, 1H), 4.88 (s, 1H), 3.85 (s, 3H), 3.83 (s, 3H), 3.82 (s, 3H); MS *m/z* 336.5 $[M+H]^+$. Anal. Calcd for $C_{16}H_{17}NO_5S$: C, 57.30; H, 5.11; N, 4.18. Found: C, 57.15; H, 5.20; N, 4.09.

3-(4-Chlorophenyl)-2,3-dihydro[1,3]dioxolo[4,5-*f*][1,2]benzisothiazole 1,1-dioxide 4g. White solid; mp 188–190 °C; IR (KBr) 3307, 3098, 2910, 1601, 1491, 1467, 1261, 1188, 1039, 899, 832, 743 cm^{-1}; ^1H NMR δ 7.37–7.43 (m, 5H), 7.02 (d, *J* = 8.1 Hz, 1H), 6.04 (d, *J* = 1.2 Hz, 1H), 5.98 (d, *J* = 1.2 Hz, 1H), 5.71 (d, *J* = 3.6 Hz, 1H), 4.86 (d, *J* = 3.6 Hz, 1H); MS *m/z* 324.4 $[M+H]^+$. Anal. Calcd for $C_{14}H_{10}ClNO_4S$: C, 51.94; H, 3.11; N, 4.33. Found: C, 51.88; H, 3.09; N, 4.29.

3-(4-Methoxyphenyl)-2,3-dihydro[1,3]dioxolo[4,5-*f*][1,2]benzisothiazole 1,1-dioxide 4h. White solid; mp 177–179 °C; IR (KBr) 3224, 3010, 1613, 1514, 1468, 1269, 1251, 1187, 1140, 1031, 897, 831, 705 cm^{-1}; ^1H NMR δ 7.39 (d, *J* = 8.1 Hz, 1H), 7.35 (dd, *J* = 6.8, 1.9 Hz, 2H), 7.01 (d, *J* = 8.1 Hz, 1H), 6.92 (dd, *J* = 6.8, 1.9 Hz, 2H), 6.01 (d, *J* = 1.2 Hz, 1H), 5.98 (d, *J* = 1.2 Hz, 1H), 5.69 (d, *J* = 4.0 Hz, 1H), 4.69 (d, *J* = 4.0 Hz, 1H), 3.83 (s, 3H); MS *m/z* 320.3 $[M+H]^+$. Anal. Calcd for $C_{15}H_{13}NO_5S$: C, 56.42; H, 4.10; N, 4.39. Found: C, 56.56; H, 4.08; N, 4.28.

3-(3,4,5-Trimethoxyphenyl)-2,3-dihydro[1,3]dioxolo[4,5-*f*][1,2]benzisothiazole 1,1-dioxide 4i. White solid; mp 213.5–215.5 °C; IR (KBr) 3266, 3007, 1593, 1518, 1472, 1299, 1139, 1036, 877, 773 cm^{-1}; ^1H NMR δ 7.39 (d, *J* = 8.1 Hz, 1H), 7.02 (d, *J* = 8.1 Hz, 1H), 6.72 (s, 2H), 6.06 (d, *J* = 1.2 Hz, 1H), 6.03 (d, *J* = 1.2 Hz, 1H), 5.67 (d, *J* = 4.0 Hz, 1H), 4.85 (d, *J* = 4.0 Hz, 1H), 3.86 (s, 9H); MS *m/z* 380.5 $[M+H]^+$. Anal. Calcd for $C_{17}H_{17}NO_7S$: C, 53.82; H, 4.52; N, 3.69. Found: C, 53.67; H, 4.56; N, 3.56.

3-(3,4-Dimethoxyphenyl)-2,3-dihydro[1,3]dioxolo[4,5-*f*][1,2]benzisothiazole 1,1-dioxide 4j. White solid; mp 175–177 °C; IR (KBr) 3265, 3006, 1593, 1518, 1472, 1298, 1265, 1139, 1035, 907, 877, 817, 758 cm^{-1}; ^1H NMR δ 7.39 (d, *J* = 8.1 Hz, 1H), 7.00-7.02 (m, 2H), 6.97 (d, *J* = 2.1 Hz, 1H), 6.87 (d, *J* = 8.1 Hz, 1H), 6.03 (d, *J* = 1.2 Hz, 1H), 5.99 (d, *J* = 1.2 Hz, 1H), 5.68 (d, *J* = 4.0 Hz, 1H), 4.79 (d, *J* = 4.0 Hz, 1H), 3.89 (s, 3H), 3.87 (s, 3H,); MS *m*/*z* 350.4 $[M+H]^+$. Anal. Calcd for $C_{16}H_{15}NO_6S$: C, 55.01; H, 4.33; N, 4.01. Found: C, 54.89; H, 4.41; N, 3.89.

3-(4-Methylphenyl)-2,3-dihydro[1,3]dioxolo[4,5-*f*][1,2]benzisothiazole 1,1-dioxide 4k. White solid; mp 218–220 °C; IR (KBr) 3426, 3213, 3095, 1498, 1471, 1287, 1139, 1037, 899, 827, 741 cm^{-1}; ^1H NMR δ 7.39 (d, *J* = 8.1 Hz, 1H), 7.33 (dd, *J* = 7.9, 4.2 Hz, 2H), 7.21 (d, *J* = 8.1 Hz, 2H), 7.01 (d, *J* = 8.1Hz, 1H), 6.01 (d, *J* = 1.2 Hz, 1H), 5.97 (d, *J* = 1.2 Hz, 1H), 5.69 (d, *J* = 4.0 Hz, 1H), 4.72 (d, *J* = 4.0 Hz, 1H), 2.38 (s, 3H); MS *m*/*z* 304.4 $[M+H]^+$. Anal. Calcd for $C_{15}H_{13}NO_4S$: C, 59.39; H, 4.32; N, 4.62. Found: C, 59.21; H, 4.29; N, 4.65.

3-(4-Fluorophenyl)-2,3-dihydro[1,3]dioxolo[4,5-*f*][1,2]benzisothiazole 1,1-dioxide 4l. White solid; mp 195.5–197.5 °C; IR (KBr) 3422, 3187, 1604, 1512, 1473, 1292, 1144, 1029, 898, 841, 820, 741 cm^{-1}; ^1H NMR δ 7.44–7.46 (m, 2H), 7.40 (d, *J* = 8.1 Hz, 1H), 7.09 (td, *J* = 8.1, 2.3 Hz, 2H), 7.02 (d, *J* = 8.1 Hz, 1H), 6.03 (d, *J* = 1.2 Hz, 1H), 5.98 (d, *J* = 1.2 Hz, 1H), 5.72 (d, *J* = 4.0 Hz, 1H), 4.82 (d, *J* = 4.0 Hz, 1H); MS *m*/*z* 308.6 $[M+H]^+$. Anal. Calcd for $C_{14}H_{10}FNO_4S$: C, 54.72; H, 3.28; N, 4.56. Found: C, 54.68; H, 3.29; N, 4.52.

3-(3-Chlorophenyl)-2,3-dihydro[1,3]dioxolo[4,5-*f*][1,2]benzisothiazole 1,1-dioxide 4m. White solid; mp 146–148 °C; IR (KBr) 3289, 1593, 1500, 1465, 1290, 1184, 1146, 1035, 899, 796 cm^{-1}; ^1H NMR δ 7.46 (d, *J* = 8.1 Hz, 1H), 7.38–7.39 (m, 1H), 7.36–7.37 (m, 1H), 7.34–7.35 (m, 2H), 7.02 (d, *J* = 8.1 Hz, 1H), 6.05 (d, *J* = 1.2 Hz, 1H), 6.00 (d, *J* = 1.2 Hz, 1H), 5.70 (d, *J* = 4.5 Hz, 1H), 4.99 (d, *J* = 4.5 Hz, 1H); MS *m*/*z* 324.4 $[M+H]^+$. Anal. Calcd for $C_{14}H_{10}ClNO_4S$: C, 51.94; H, 3.11; N, 4.33. Found: C, 51.78; H, 3.09; N, 4.36.

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