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Novel preparation of 2,1-benzothiazine derivatives from sulfonamides with [hydroxy(tosyloxy)iodo]arenes

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Cyclization of sulfonamides bearing an aromatic ring at the β-position with various organohypervalent iodine compounds was carried out to form the corresponding 2,1-benzothiazine derivatives. Among them, the cyclization effectively proceeded with [hydroxy(tosyloxy)iodo]arenes through ionic pathways. The same treatment of a sulfonamide bearing a 4-methoxyphenyl group at the β-position generated a spiro compound.

Introduction

It is well known that most sulfonamides possess biological activities.[1] In particular, cyclic sulfonamides (sultams) are important as therapeutic compounds[2] and chiral auxiliaries.[3] Among them, 3,4-dihydro-2,1-benzothiazine 2,2-dioxide derivatives (benzosultams) have potent biological activities such as lipoxygenase inhibition and are used as drugs for treating heart diseases (Fig. 1). These broad biological activities are the reason why profound attention has been paid to the synthesis of the 2,1-benzothiazine skeleton.[4] Today, four established methods for the construction of the 3,4-dihydro-2,1-benzothiazine 2,2-dioxide skeleton are known, *i.e.*, cyclization of *N*-benzyl-*N*-methanesulfonyl(*o*-chloromethyl)aniline with NaH,[5a] pyrolysis of β-arylethanesulfonyl azides,[5b] cyclization of *N*-phenylsulfamoylacetic acid with polyphosphoric acid (PPA) and subsequent reduction of the carbonyl group,[5c] and cyclization of 2-(*o*-aminophenyl)ethanesulfonic acid with $POCl_3$.[5d]

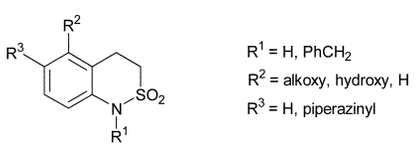

Fig. 1 3,4-Dihydro-2,1-benzothiazine 2,2-dioxide derivatives.

However, these methods require many steps from commercially available materials and quite acidic or basic conditions; moreover, the yields of the cyclized products are generally not very high. So we planned to develop a new synthetic method for the 2,1-benzothiazine 2,2-dioxide skeleton.

In our laboratory, the novel preparation of heterocyclic compounds with hypervalent iodine reagents and iodine under photolytic conditions has been studied, under mild conditions with clean transformation and low toxicity.[6] Recently, we reported a new preparative method for 3,4-dihydro-2,1-benzothiazine 2,2-dioxides from *N*-alkyl-2-(aryl)ethanesulfonamides *via* a radical pathway, with (diacetoxyiodo)arenes in the presence of iodine under photochemical conditions.[7] Using this method, the six-membered benzosultams were obtained in high yields. However, five- and seven-membered benzosultams were not obtained; moreover, deprotection to free the NH group from the *N*-alkyl group in the six-membered benzosultams was rather difficult.

We planned to prepare the 3,4-dihydro-2,1-benzothiazine skeleton *via* an ionic pathway with hypervalent iodine compounds due to easy deprotection to free the NH group and its synthetic application to five- and seven-membered benzosultams. Cyclization of *N*-methoxypropionamide to its aromatic ring with [bis(trifluoroacetoxy)iodo]benzene is known to form benzolactams *via* *N*-acylnitrenium ions.[8]

Results and discussion

At first, when *N*-methoxy-2-(phenyl)ethanesulfonamide **1a** was treated with a hypervalent iodine reagent (1.1 eq.) in acetonitrile under an argon atmosphere at 0 °C–r.t. for 20 min, *N*-methoxy-3,4-dihydro-2,1-benzothiazine 2,2-dioxide **2a** was obtained. The effect of different hypervalent iodine reagents on the cyclization of compound **1a** was examined as shown in Table 1. [Hydroxy(tosyloxy)iodo]benzene gave compound **2a** in the best yield (86%) (Entry 5), and 3-trifluoromethyl-1-[hydroxy(tosyloxy)iodo]benzene also worked well (Entry 6). Thus, an increase of the electrophilic character of the iodine of the hypervalent iodine reagent accelerates the cyclization. When iodosobenzene was used, compound **2a** was obtained only in 8% yield (Entry 3). However, compound **2a** was obtained in 51% yield by the addition of boron trifluoride etherate as a Lewis acid (Entry 4). Here, boron trifluoride etherate accelerates the solubility and electrophilicity of iodosobenzene in acetonitrile. Moreover, in order to increase the electrophilic character of iodine, hypervalent iodine reagents bearing an electron-withdrawing group such as the *p*-chlorobenzenesulfonyloxy group (Entry 7) and the *m*-nitrobenzenesulfonyloxy group (Entry 8) were used. However, no increase in the yield of compound **2a** was observed.

The effect of temperature was studied as shown in Table 2. When the reaction was carried out under stirring at 0 °C to r.t. for 20 min, the best yield was obtained (Entry 4). It is not effective to dilute the concentration of substrate **1a**, to prevent the intermolecular coupling reaction.

Furthermore, the solvent effect in the present cyclization was examined as shown in Table 3, and the yield roughly depends on the relative permittivity (ε). Thus, the cyclization proceeded effectively in acetonitrile (Entry 1). In 1,2-dichloroethane or benzene, the cyclization also proceeded in moderate to good yields (Entries 2, 3). However, the cyclization did not work effectively in ethyl acetate or chloroform (Entries 4, 5).

On the basis of the results shown in Tables 1–3, the formation of 3,4-dihydro-2,1-benzothiazine 2,2-dioxides with other *N*-methoxy-2-(aryl)ethanesulfonamides under the best conditions was carried out as shown in Table 4. Here, with respect to

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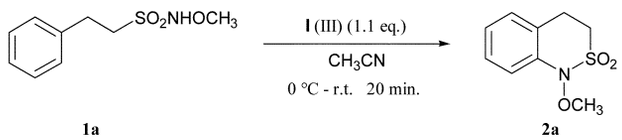
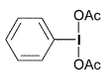
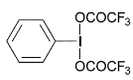
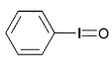
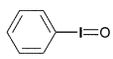
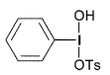
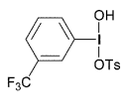
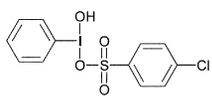
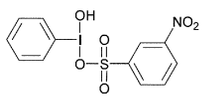
Table 1 Cyclization of *N*-methoxy-2-(phenyl)ethanesulfonamide: effect of hypervalent iodine reagents

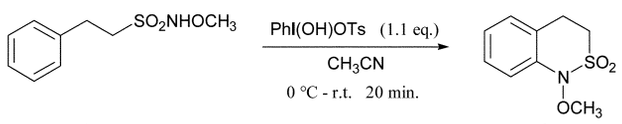


1a **2a**

Entry	I (III)	Yield (%)
1		54
2		71
3		8
4[a]		51
5		86
6		81
7		71
8		73

[a] $BF_3{\cdot}OEt_2$ (2.0 eq.) was added.

Table 2 Effect of temperature

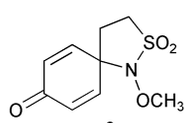


1a **2a**

	Conditions		
Entry	Temp./°C	Time/min	Yield (%)
1	60–65	5	65
2	0	20	65
3	10–15	20	84
4	0–r.t.	20	86
5[a]	0–r.t.	20	52

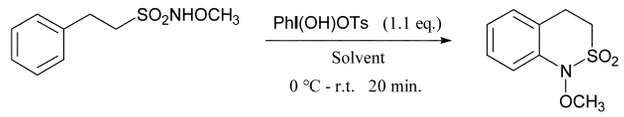
[a] Reaction was carried out under a half concentration of compound **1a**.

the *p*-substituent effect of the aromatic ring in *N*-methoxy-2-(aryl)ethanesulfonamide, an electron-withdrawing group such as a *p*-chloro (**c**) and *p*-fluoro (**d**) group reduced the formation of benzosultams. This result suggests that this cyclization onto the aromatic ring proceeds by the electrophilic *N*-sulfonylnitrenium species. *N*-Methoxy-2-(aryl)ethanesulfonamide bearing the *p*-methoxy (**e**) group gave the spirocyclized product (**3**) which is formed from the reaction at the *ipso*-position, instead of the *o*-position in the aromatic ring.



3

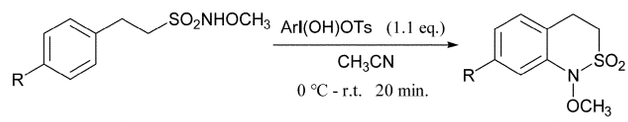
Table 3 Effect of solvent



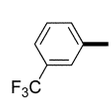
1a **2a**

Entry	Solvent	Yield (%)	ε
1	CH_3CN	86	37.5
2	$ClCH_2CH_2Cl$	73	10.4
3	C_6H_6	62	2.3
4	CH_3CO_2Et	29	6.0
5	$CHCl_3$	18	4.7

Table 4 Reactivity of *N*-methoxy-2-(aryl)ethanesulfonamides **1a**–**1e**



1 **2**

Entry	Substrate	R	Ar	Yield (%)
1	**1a**	H		86
2	**1b**	CH_3		85
3	**1c**	Cl		52
4	**1d**	F		44
5	**1e**	OCH_3		0 (64)[a]
6	**1a**	H		81
7	**1b**	CH_3		78
8	**1c**	Cl		51
9	**1d**	F		45
10	**1e**	OCH_3		0 (66)[a]

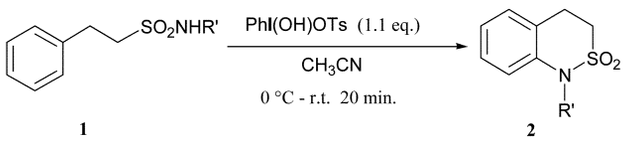
[a] Compound **3** was obtained.

The effect of *N*-substituted sulfonamides on the present cyclization was examined as shown in Table 5. Here, the *N*-methoxy (**a**) group gave the cyclized compound. However, *N*-free (**f**), *N*-methyl (**g**), and *N*-acetyl (**h**) groups did not give the cyclization product at all under any conditions, and the starting materials were recovered quantitatively. These results indicated that the *N*-methoxy group plays an important role in the formation of a hypervalent iodine-binding *N*-sulfonylnitrenium ion intermediate. The yield of compound **2**, ^{1}H NMR chemical shifts of the NH and CH_2SO_2 groups, and ^{13}C NMR chemical shifts of the CH_2SO_2 group in starting material **1**, are shown in Table 5. A good relationship between the yield and the chemical shifts was not observed. However, we believe this reactivity probably depends on the acidity of the sulfonamide NH group in compound **1**.

N-Methoxy-2-(α-naphthyl)ethanesulfonamide **4** gave the corresponding 3,4-dihydro-2,1-naphthothiazine 2,2-dioxide **5** in high yield, under the same reaction conditions. When the present cyclization was carried out with *N*-methoxy(phenyl)-methanesulfonamide **6**, *N*-methoxy-1,3-dihydro-2,1-benzisothiazole 2,2-dioxide **7** was obtained in 52% yield. Similarly, *N*-methoxy-1,3,4,5-tetrahydro-2,1-benzothiazepine 2,2-dioxide **9** was obtained in 37% yield from *N*-methoxy-3-(phenyl)-propanesulfonamide **8**. Compounds **7** and **9** could not be obtained with the radical reaction method.[7] Moreover, *N*-methoxy-2-(indolyl)ethanesulfonamide **10** gave the corresponding cyclized product **11** in good yield (Scheme 1).

On the basis of these results, a plausible reaction mechanism for the formation of the 3,4-dihydro-2,1-benzothiazine 2,2-

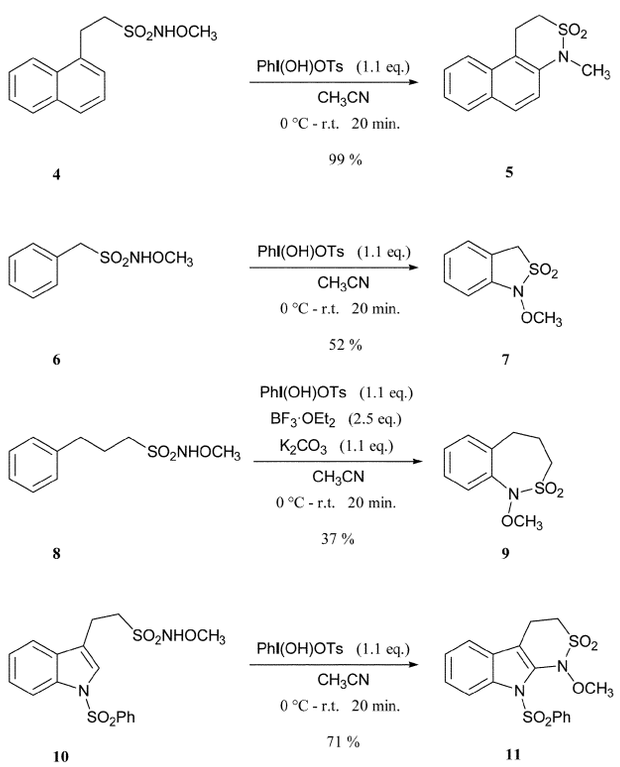
Table 5 Reactivity of *N*-substituted 2-(phenyl)ethanesulfonamides



1 2

				NMR ($CDCl_3$, TMS) of compound **1**		
				^{1}H NMR (ppm)		^{13}C NMR (ppm)
Entry	Substrate	R′	Yield (%)	NH	CH_2SO_2	CH_2SO_2
1	**1a**	OCH_3	86	6.64	3.51	49.84
2	**1f**	H	0 (98)[a]	4.51	3.42	56.50
3	**1g**	CH_3	0 (91)[a]	4.01	3.31	52.25
4	**1h**	$COCH_3$	0 (97)[a]	8.34	3.75	53.85

[a] Starting material was recovered.



Scheme 1 Formation of benzosultam derivatives.

dioxide skeleton is shown in Scheme 2. *N*-Methoxy-2-(aryl)-ethanesulfonamide **1** reacts on the polarized iodine atom of [hydroxy(tosyloxy)iodo]benzene to give intermediate **A** in which the electron-deficient nitrogen atom then reacts directly at the *o*-position of the aromatic ring with the side chain to give a 3,4-dihydro-2,1-benzothiazine 2,2-dioxide skeleton. Another plausible reaction mechanism is the formation of spiro intermediate **B** through an initial *ipso* attack, followed by a 1,2-shift of the *N*-methoxyamide group to generate a 3,4-dihydro-2,1-benzothiazine 2,2-dioxide skeleton. On the same treatment, *N*-methoxy-2-(aryl)ethanesulfonamide bearing a *p*-methoxy group at the aromatic ring undergoes spirocyclization at the *ipso*-position of the aromatic ring, together with formation of methanol.

In order to support the formation of methanol, *N*-methoxy-2-(4-benzyloxyphenyl)ethanesulfonamide **1i** was treated under the same conditions to form **3** and benzyl alcohol **12** in 62% and 92% yields, respectively (Scheme 3).

Finally, *N*-methoxy-3,4-dihydro-2,1-benzothiazine 2,2-dioxide was easily reduced to give *N*-free 3,4-dihydro-2,1-benzothiazine 2,2-dioxide in 99% yield by treatment with samarium diiodide in THF. Similarly, *N*-methoxy-7-methyl-3,4-dihydro-2,1-benzothiazine 2,2-dioxide was also reduced quantitatively under the same conditions.

In conclusion, the present cyclization of *N*-methoxy-2-(aryl)ethanesulfonamides with [hydroxy(tosyloxy)iodo]arenes through ionic pathways is very useful for the preparation of 3,4-dihydro-2,1-benzothiazine 2,2-dioxide derivatives, which bear potent biological activity, in good yields. Moreover, *N*-methoxy-2-(aryl)ethanesulfonamide bearing a 4-methoxy group at the aromatic ring generates a spiro compound. The *N*-methoxy sultams formed can be easily reduced to the corresponding *N*-free sultams with samarium diiodide.

Experimental

General

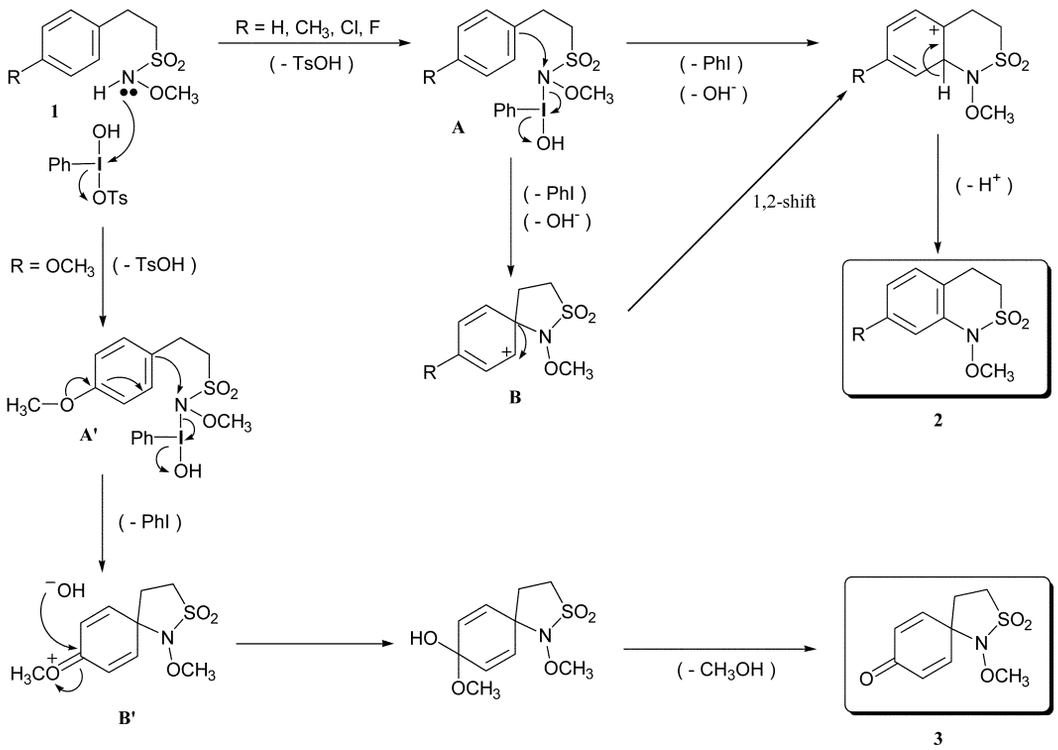
^{1}H NMR and ^{13}C NMR spectra were obtained with JEOL-JNM-LA-400, JEOL-JNM-LA-400s, and JEOL-JNM-LA-500 spectrometers. Chemical shifts are expressed in ppm downfield from TMS in δ units; quintets are represented as p. Mass spectra were recorded on JEOL-HX-110 and JEOL-JMS-AT II 15 spectrometers. Melting points were determined on Yamato melting points apparatus Model MP-21. Silica Gel 60 (Kanto Kagaku Co) was used for column chromatography, and Wakogel B-5F was used for preparative TLC.

Materials

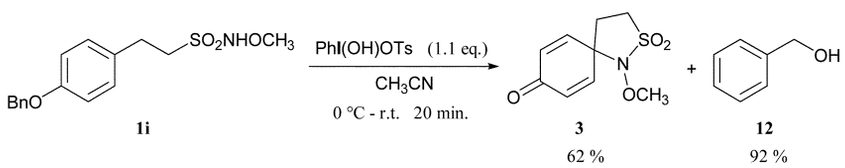
(Diacetoxyiodo)benzene, [bis(trifluoroacetoxy)iodo]benzene and iodosobenzene are commercially available. The other hypervalent iodine compounds were prepared based on the literature methods.[9] Most sulfonamides were prepared by the reaction of sulfonyl chlorides with *O*-methylhydroxylamine hydrochloride. Sulfonyl chlorides were prepared from the corresponding sodium sulfonates, which were prepared from the reaction of alkyl bromides and sodium sulfite.[10]

General procedure for the conversion of *N*-methoxy-2-(aryl)-ethanesulfonamides to the corresponding 3,4-dihydro-2,1-benzothiazine 2,2-dioxide

Hypervalent iodine compound (0.55 mmol) was added to a solution of *N*-methoxy-2-(aryl)ethanesulfonamide (0.5 mmol) in acetonitrile (3 mL). The mixture was stirred at 0 °C to r.t. for 20 min under an argon atmosphere. After the reaction, the mixture was poured into a saturated aqueous sodium sulfite solution and extracted with chloroform three times. The organic layer was dried over sodium sulfate. After removal of the solvent under reduced pressure, the residue was treated with preparative TLC on silica gel using a mixture of hexane and ethyl acetate (3:1) as an eluent.



Scheme 2 Plausible reaction mechanism.



Scheme 3 Support for the formation of alcohol.

N-Methoxy-3,4-dihydro-2,1-benzothiazine 2,2-dioxide (2a)

Mp 104.0–106.0 °C; IR (KBr) 3000, 2950, 2815, 1580, 1480, 1360, 1170 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.42 (t, J = 6.4 Hz, 2H), 3.50 (td, J = 6.4, 1.5 Hz, 2H), 4.08 (s, 3H), 7.20–7.23 (m, 2H), 7.31–7.34 (m, 1H), 7.36–7.40 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 27.86 (s), 40.20 (s), 65.57 (p), 126.72 (q), 127.88 (t), 128.03 (t), 128.90 (t), 129.41 (t), 141.88 (q); MS (EI) M^+ 213. Anal. Calcd for $C_9H_{11}NO_3S$: C, 50.69; H, 5.20; N, 6.57. Found: C, 50.76; H, 5.32; N, 6.58%.

N-Methoxy-7-methyl-3,4-dihydro-2,1-benzothiazine 2,2-dioxide (2b)

Mp 119.0–121.0 °C; IR (KBr) 3000, 2950, 2815, 1620, 1500, 1360, 1170 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$) δ 2.36 (s, 3H), 3.36 (t, J = 6.6 Hz, 2H), 3.47 (t, J = 6.6 Hz, 2H), 4.07 (s, 3H), 7.08 (d, J = 7.9 Hz, 1H), 7.12 (dd, J = 7.6, 1.6 Hz, 1H), 7.18 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.99 (p), 27.56 (s), 40.20 (s), 65.68 (p), 123.61 (q), 128.27 (t), 129.32 (t), 130.07 (t), 138.22 (q), 141.63 (q); MS (EI) M^+ 227. Anal. Calcd for $C_{10}H_{13}NO_3S$: C, 52.85; H, 5.77; N, 6.16. Found: C, 52.80; H, 5.69; N, 6.14%.

N-Methoxy-7-chloro-3,4-dihydro-2,1-benzothiazine 2,2-dioxide (2c)

Mp 123.0–125.0 °C; IR (KBr) 3000, 2950, 2815, 1600, 1480, 1360, 1160 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.37 (t, J = 6.7 Hz, 2H), 3.48 (t, J = 6.5 Hz, 2H), 4.08 (s, 3H), 7.14 (d, J = 8.2 Hz, 1H), 7.27 (dd, J = 8.5, 2.0 Hz, 1H), 7.36 (d, J = 2.1 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 27.55 (s), 40.42 (s), 65.95 (p), 125.08 (q), 127.14 (t), 128.92 (t), 130.60 (t), 133.53 (q), 142.90 (q); MS (EI) M^+ 247. Anal. Calcd for $C_9H_{10}ClNO_3S$: C, 43.64; H, 4.07; N, 5.65. Found: C, 43.39; H, 4.08; N, 5.52%.

N-Methoxy-7-fluoro-3,4-dihydro-2,1-benzothiazine 2,2-dioxide (2d)

Mp 76.0–78.0 °C; IR (KBr) 3000, 2950, 2820, 1600, 1490, 1360, 1170 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.37 (t, J = 6.4 Hz, 2H), 3.47 (td, J = 6.4, 1.3 Hz, 2H), 4.07 (s, 3H), 7.02 (td, J = 8.2, 2.7 Hz, 1H), 7.08 (dd, J = 8.8, 2.7 Hz, 1H), 7.17 (dd, J = 8.6, 6.0 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.22 (s), 40.48 (s), 65.83 (p), 113.52 (t, J_{C-F} = 23.8 Hz), 115.96 (t, J_{C-F} = 22.1 Hz), 122.14 (q, J_{C-F} = 4.2 Hz), 130.72 (t, J_{C-F} = 8.2 Hz), 142.84 (q, J_{C-F} = 9.8 Hz), 161.64 (q, J_{C-F} = 247.5 Hz); MS (EI) M^+ 231. Anal. Calcd for $C_9H_{10}FNO_3S$: C, 46.75; H, 4.36; N, 6.06. Found: C, 46.94; H, 4.41; N, 5.89%.

1-Methoxy-2,2-dioxo-2λ^6-thia-1-aza-spiro[4.5]deca-6,9-dien-8-one (3)

Mp 133.0–135.0 °C; IR (KBr) 2980, 2950, 2830, 1680, 1610, 1400, 1330, 1160 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.44 (t, J = 8.0 Hz, 2H), 3.44 (t, J = 7.9 Hz, 2H), 3.81 (s, 3H), 6.39 (d, J = 10.1 Hz, 2H), 7.05 (d, J = 10.4 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 27.19 (s), 43.15 (s), 61.79 (q), 66.62 (p), 131.12 (t), 145.74 (t), 184.14 (q); MS (EI) M^+ 229. Anal. Calcd for $C_9H_{11}NO_4S$: C, 47.15; H, 4.84; N, 6.11. Found: C, 47.16; H, 4.68; N, 6.07%.

N-Methoxy-3,4-dihydro-2,1-naphthothiazine 2,2-dioxide (5)

Mp 137.0–139.0 °C; IR (KBr) 2980, 2950, 2820, 1600, 1510, 1360, 1170 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.64 (t, J = 6.8 Hz, 2H), 3.77 (t, J = 6.7 Hz, 2H), 4.18 (s, 3H), 7.48 (d, J = 8.7 Hz, 1H), 7.54–7.62 (m, 2H), 7.81–7.62 (m, 2H), 7.95 (d, J = 7.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 25.48 (s), 38.99 (s), 66.06 (p), 121.99 (q), 123.29 (t), 125.64 (t), 126.92 (t),

127.33 (t), 128.76 (t), 129.15 (t), 131.29 (q), 132.84 (q), 139.30 (q); MS (EI) M^+ 263. Anal. Calcd for $C_{13}H_{13}NO_3S$: C, 59.30; H, 4.98; N, 5.32. Found: C, 59.35; H, 4.89; N, 5.29%.

N-Methoxy-1,3-dihydrobenzisothiazole 2,2-dioxide (7)

Oil; IR (neat) 3000, 2940, 2820, 1590, 1500, 1360, 1160 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 4.14 (s, 3H), 4.34 (s, 2H), 7.22 (m, 2H), 7.39 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 49.97 (s), 65.88 (p), 120.02 (q), 125.67 (t), 126.23 (t), 128.81 (t), 129.79 (t), 142.39 (q); HRMS (FAB) Found M^+ 199.0288. Calcd for $C_8H_9NO_3S$ M 199.0303.

N-Methoxy-1,3,4,5-tetrahydro-2,1-benzothiazepine 2,2-dioxide (9)

Mp 127.0–129.0 °C; IR (KBr) 2980, 2860, 2825, 1600, 1490, 1360, 1160 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.16 (t, J = 5.8 Hz, 2H), 3.04 (t, J = 5.1 Hz, 2H), 3.71 (t, J = 6.1 Hz, 2H), 3.89 (s, 3H) 7.17 (td, J = 7.9, 1.5 Hz, 1H), 7.24–7.34 (m, 2H), 7.43 (dd, J = 3.8, 1.7 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.96 (s), 32.84 (s), 51.40 (s), 64.37 (p), 127.88 (t), 130.86 (t), 131.77 (t), 132.39 (t), 138.45 (q), 141.73 (q); MS (EI) M^+ 227. Anal. Calcd for $C_{10}H_{13}NO_3S$: C, 52.85; H, 5.77; N, 6.16. Found: C, 52.52; H, 5.54; N, 6.14%.

1-Methoxy-9-benzenesulfonyl-3,4-dihydro-2,1-indolothiazine 2,2-dioxide (11)

Mp 162.0 °C (decomp.); IR (KBr) 3100, 2990, 2950, 2820, 1600, 1480, 1360, 1160 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.51 (ddd, J = 13.3, 9.9, 8.4 Hz, 1H), 2.78 (s, 3H), 2.98 (ddd, J = 13.4, 8.9, 3.0 Hz, 1H), 3.37–3.48 (m, 2H), 6.21 (s, 1H), 7.13 (td, J = 7.5, 1.0 Hz, 1H), 7.32 (dd, J = 7.6, 0.8 Hz, 1H), 7.39–7.49 (m, 2H), 7.56 (tt, J = 7.4, 1.3 Hz, 1H), 7.66 (d, J = 8.2 Hz, 1H), 7.89 (dt, J = 7.3, 1.3 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.44 (s), 46.48 (s), 64.91 (p), 73.30 (q), 85.41 (q), 114.42 (t), 124.43 (t), 125.62 (t), 127.47 (t), 128.11 (q), 129.06 (t), 131.40 (t), 133.48 (t), 137.60 (q), 141.03 (q); MS (FAB) M^+ + 1 393. Anal. Calcd for $C_{17}H_{16}N_2O_5S_2$: C, 52.03; H, 4.11; N, 7.14. Found: C, 51.91; H, 4.02; N, 7.05%.

3,4-Dihydro-2,1-benzothiazine 2,2-dioxide

Mp 150.0–152.0 °C (lit.[5d] 151–153 °C); ^1H NMR (400 MHz, $CDCl_3$) δ 3.32 (t, J = 6.9 Hz, 2H), 3.49 (t, J = 6.8, 2H), 6.45 (s, 1H), 6.75 (dd, J = 8.1, 1.1 Hz, 1H), 7.05 (td, J = 7.5, 1.1 Hz, 1H), 7.17–7.23 (m, 2H)

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