

CYCLIZATION REACTIONS OF 2-METHYLBENZENESULFONAMIDES USING *N,N*-DIMETHYLCARBAMOYL CHLORIDE, *N,N*-DIMETHYLTHIOCARBAMOYL CHLORIDE, AND *N,N*-DIMETHYLSULFAMOYL CHLORIDE

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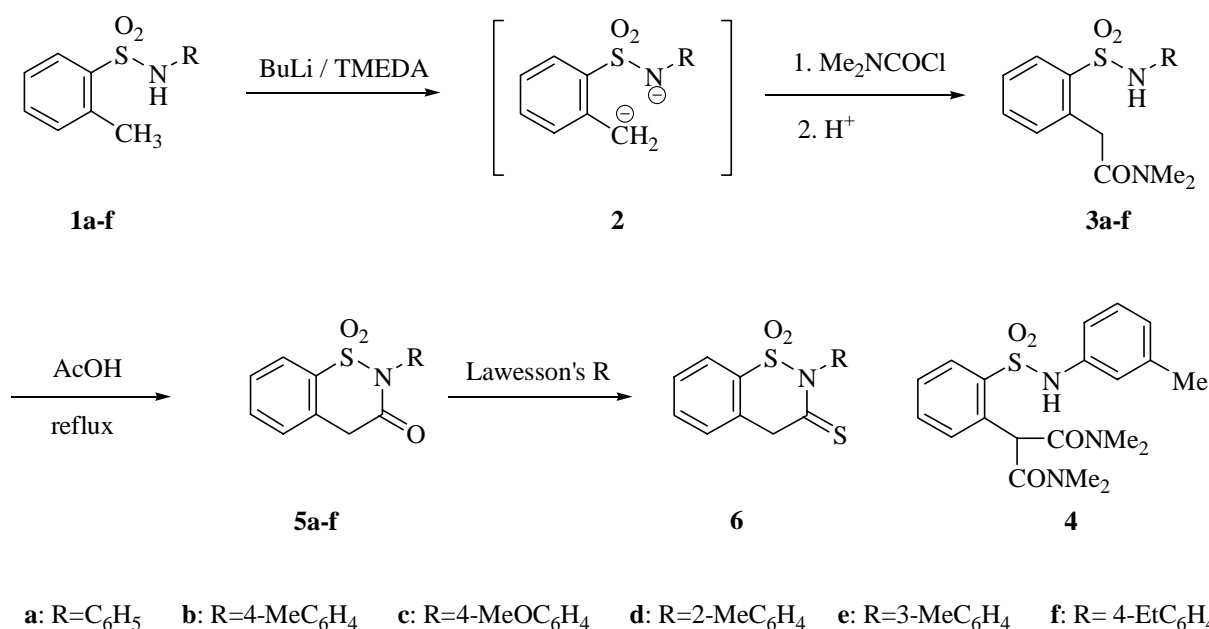
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Abstract-Generation of C,N-dianions (**2**) of 2-methylbenzenesulfonamides (**1**) followed by addition of *N,N*-dimethylcarbamoyle chloride gave 2-(*N,N*-dimethylcarbamoyle)-methylbenzenesulfonamides (**3**), which were cyclized to 3,4-dihydro-2*H*-1,2-benzo[*e*]thiazin-3-one 1,1-dioxides (**5**). 3-Dimethylamino-2*H*-1,2-benzo[*e*]thiazine 1,1-dioxides (**7**) were obtained in one step by the reaction of **2** with *N,N*-dimethylthiocarbamoyle chloride. On the other hand, the reaction of **2** with *N,N*-dimethylsulfamoyle chloride yielded 2,2'-ethylene-bis(benzenesulfonamide)s (**10**) and / or 2,3-dihydro-1,2-benzothiazole 1,1-dioxides (**11**).

Ortho-lithiation of benzenesulfonamides was first found by Watanabe *et al.*¹ and has been used for the synthesis of heterocycles.² On the other hand, the lithiation of 2-methylbenzenesulfonamides (**1**) generates C, N-dianions (**2**), where hydrogen on the methyl group instead of the *ortho*-hydrogen of the benzene ring is abstracted.³ This C, N-dilithiation reaction was applied to the synthesis of 2-substituted 3-phenyl-1,2-benzo[*e*]thiazine 1,1-dioxides by reaction with benzonitrile⁴ and 2-substituted 3,4-dihydro-1,2-benzo[*e*]thiazin-3-one 1,1-dioxides by reaction with carbon dioxide followed by dehydration.⁵ Recently, the synthetic utility of 2-methylbenzenesulfonamides has become of interest again; various saccharin derivatives have been prepared by reaction of 2-methylbenzenesulfonamides with

(diacetoxyiodo)arene in the presence of iodine under irradiation with a tungsten lamp.⁶ 2-Substituted 2*H*-1,2-benzo[*e*]thiazine 1,1-dioxides have been obtained by lithiation/cyclization reaction of *N*-acyl-2-methylbenzenesulfonamides.⁷ Quenching of lithiated *N*-Boc-2-methylbenzenesulfonamides with ketones followed by dehydration gave 3,3-disubstituted 2*H*-1,2-benzo[*e*]thiazine 1,1-dioxides.⁸ As a part of our research program on heterocyclic synthesis using sulfonamides,⁹ we studied the reactivity of **2** towards a series of acid chlorides such as *N,N*-dimethylcarbamoyl chloride, *N,N*-dimethylthiocarbamoyl chloride, and *N,N*-dimethylsulfamoyl chloride. It appears that these acid chlorides show different reactivities to **2** and the results are presented in this paper.

Scheme 1

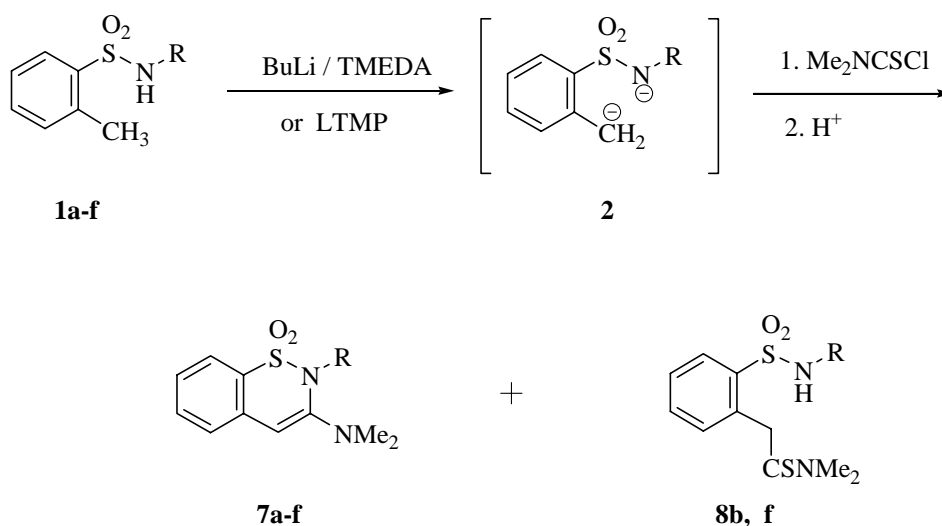


N-Aryl-2-methylbenzenesulfonamides (**1**) were prepared from the readily available 2-methylbenzenesulfonyl chlorides and *N*-substituted amines in the usual manner. In order to obtain the cyclized product 3,4-dihydro-2*H*-1,2-benzo[*e*]thiazine 1,1-dioxides (**5**), C, N-dianions (**2**) generated in THF at -75 °C on treatment of **1** with butyllithium in the presence of *N,N,N',N'*-tetramethylethylenediamine (TMEDA) were subjected to reaction with *N,N*-dimethylcarbamoyl chloride. However, 2-[*N*-arylsulfamoylphenyl]-*N,N*-dimethylacetamides (**3**) were obtained in 40-65% yields (Scheme 1). In the case of *N*-3-methylphenylsulfamoyl derivative (**1e**), propanediamide (**4**) (24%) was formed as a by-product (Tables 1 and 2). When *N*-alkyl-2-methylbenzenesulfonamides were used as the starting sulfonamides and treated in a similar manner, the products were sometimes difficult to purify, and therefore only *N*-arylsulfamoyl products were identified. In the NMR spectra, the two methyl groups of **3** appeared in two singlet absorptions characteristic of an amide group. The cyclization of **3** to **5** was achieved in 52-71% yields under the

conditions of refluxing a mixture of **3** in acetic acid, that were employed for the cyclization of *N,N*-diethyl-2-sulfamoylbenzamide to benzisothiazol-3-one 1,1-dioxides (saccharin).¹⁰

In some trials to prepare derivatives of **5**, a thione (**6**) (R= 4-MeOC₆H₄) was obtained in 65% yield on treatment with Lawesson's reagent. The thione (**6**) seems to be produced on treatment of **1** with *N,N*-dimethylthiocarbamoyl chloride instead of *N,N*-dimethylcarbamoyl chloride. However, lithiation of **1** using BuLi / TMEDA or LTMP (BuLi / 2,2,4,4-tetramethylpiperidine) followed by addition of *N,N*-dimethylthiocarbamoyl chloride furnished the cyclized products 2-aryl-3-dimethylamino-2*H*-1,2-benzo[*e*]thiazines (**7a-f**) in one step in 15-32% yields, accompanied by the formation of the by-products (**8b**) and (**f**) (Scheme 2, and Tables 3 and 4). 1,2-Benzo[*e*]thiazine 1,1-dioxide derivatives are important because of their diuretic, anti-inflammatory, and anti-bacterial activities¹¹ and potential prodrugs of nitroxyl.¹² Interesting chemical behavior in these heterocycles is a ring-enlargement reaction with 3-amino-2*H*-azirine to form 1,2,5-benzothiadiazonin-6-one 1,1-dioxides¹³ and an electrophilic fluorination reaction of an *N*-fluoro derivative to carbanions.¹⁴

Scheme 2

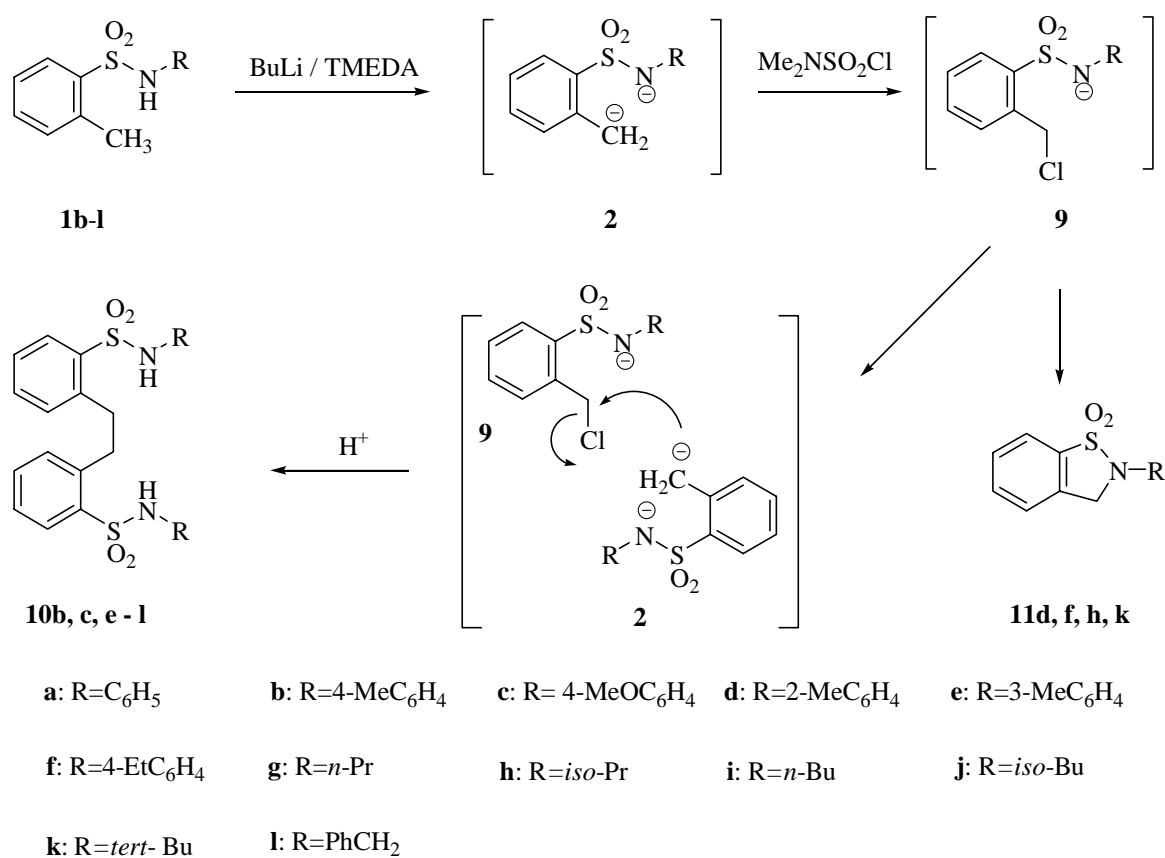


a: R=C₆H₅ **b:** R=4-MeC₆H₄ **c:** R= 4-MeOC₆H₄ **d:** R=2-MeC₆H₄ **e:** R=3-MeC₆H₄ **f:** R=4-EtC₆H₄

In contrast to the reactions described above, the reaction of **2** with *N,N*-dimethylsulfamoyl chloride proceeded in a different manner (Scheme 3) and gave two kinds of products, 2,2'-ethylene-bis(benzensulfonamide)s (**10b**, **c**, **e-l**), in 5-60% yields, and / or 2-substituted 1,2-benzothiadiazole 1,1-dioxides (**11d**, **f**, **h**, **k**), in 6-33% yields (Table 5). These structures were confirmed unequivocally by spectral and analytical data (Table 6). The dimer (**10a**) corresponding to **1a** was not included in Tables 5 and 6 because of the difficulty in preparing an analytically pure sample. It was shown

that the use of other sulfonyl chlorides such as benzenesulfonyl chloride or trifluoromethanesulfonyl chloride instead of *N,N*-dimethylsulfonyl chloride gave the same products but in lower yields. Carbanions are reported to be chlorinated by *N,N*-dimethylsulfamoyl chloride.¹⁵ Furthermore, dimerization of β -keto sulfone dianions in the presence of iodine is known to give 1,4-dicarbonyl compounds (oxidative coupling),¹⁶ and lithiated β -aminoalkyl sulfones were shown to form dimers in the presence of 1,2-diiodoethane.¹⁷ In view of these reports, the formation of the dimer products (**10**)

Scheme 3



may be explained by the initial formation of chlorinated intermediates (**9**) followed by the intermolecular attack of **2** on **9**. On the other hand, the intramolecular attack of the N-anion of **9** on the chloromethyl group would lead to the formation of **11**. This mechanism is supported by the following experiment in **1f**. As the molar ratio of **1f**: BuLi : Me₂NSO₂Cl was changed from 1 : 2 : 1 to 1 : 2 : 2, the yield of **11f** increased from 6% to 30% with a decrease of the yield of **10f** from 48% to 1.3%. This indicates that as the results of an increased amount of **9f** and a relatively decreased amount of **2f**, cyclization in **9f** would prefer to intermolecular attack of **2f** on **9f**.

In summary, we have developed two new routes to 1,2-benzo[e]thiazine 1,1-dioxides from 2-methylbenzenesulfonamides and shown the first dimerization reaction of 2-methylbenzenesulfonamides accompanied by the intramolecular cyclization to 1,2-benzothiazole 1,1-dioxides.

Table 1. Physical Properties of Compounds (3 and 5)

Compound	Yield (%)	mp (°C)	Calcd			Found		
			C	H	N	C	H	N
3a	51	161-163	60.36	5.70	8.80	60.51	5.71	8.63
3b	65	182-184	61.42	6.06	8.43	61.35	6.13	8.42
3c	47	148-149	58.60	5.79	8.04	58.39	5.81	8.09
3d	53	133-135	61.42	6.06	8.43	61.45	6.11	8.52
3e	40	132-133	61.42	6.06	8.43	61.54	6.08	8.59
3f	63	132-134	62.40	6.40	8.09	62.20	6.47	7.93
5a	61	117-120 (lit., ¹⁸ mp 122-123 °C)						
5b	60	188-190	62.70	4.56	4.87	62.35	4.68	4.91
5c	60	165-167	59.40	4.32	4.62	59.06	4.44	4.65
5d	71	129-131	62.70	4.56	4.87	62.83	4.71	4.96
5e	68	156-158	62.70	4.56	4.87	62.42	4.68	5.00
5f	52	110-111	63.77	5.02	4.65	63.60	5.16	4.74

EXPERIMENTAL

Melting points were determined with MRK MEL-TEMP II and are uncorrected. The IR spectra were measured on JASCO A-102 and JASCO FT/IR-420 spectrophotometers. MS and ¹H-NMR spectra were taken with JEOL JMS DX-300 spectrometer and JEOL GSX-400 spectrometer, respectively. Microanalyses were performed with YANACO CHN-CODER MT-5.

2-[2-(*N*-Arylsulfamoyl)]phenyl-*N,N*-dimethylacetamides (**3**). General procedure.

To a solution of **1** (10.0 mmol) in dry THF (30 mL) cooled at – 75 °C under nitrogen atmosphere was slowly added a solution of butyllithium in hexane (1.52 M) (13.5 mL, 20.5 mmol) during more than 15 min and then TMEDA (3.1 mL, 20.5 mmol). The mixture was stirred at rt for 30 min and cooled to – 75 °C, and then dropwise added with *N,N*-dimethylcarbamoyl chloride (0.93 mL, 10.5 mmol). After stirring at rt for 2 days, the mixture was neutralized with 10% hydrochloric acid (15 mL) and then diluted with water (100 mL) to give precipitates, which were collected by

Table 2. Spectral Data of Compounds (3 and 5)

	MS, m/z (%)	IR (KBr)	$^1\text{H-NMR}$ (CDCl_3),
3a	318 (M^+ , 8), 226 (100), 72 (30)	3100, 1630, 1595, 1490, 1340, 1170	3.06 (s, 3H), 3.18 (s, 3H), 4.23 (s, 2H), 7.10-7.44(m,9H), 8.33 (s, 1H)
3b	332 (M^+ , 6), 226 (100), 72 (34)	3150, 1630, 1515, 1480, 1400, 1340	2.27(s,3H),3.05(s,3H),3.19(s,3H),4.22(s,2H),7.13-7.44 (m, 8H), 8.19 (s, 1H)
3c	348 (M^+ 15), 226 (100), 123 (66),	3200, 1635, 1515, 1470, 1405, 1340	3.04(s,3H),3.19(s,3H),3.76(s,3H),4.23(s,2H),6.72-7.44 (m, 8H), 8.10 (s, 1H)
3d	332 (M^+ , 4), 226 (100), 106 (72)	3210, 1640, 1495, 1410, 1400, 1330	2.13(s,3H),3.04(s,3H),3.19(s,3H),4.22(s,2H),7.01-7.49 (m, 8H), 8.08 (s, 1H)
3e	332 (M^+ , 6), 226 (100), 72 (31)	3160, 1625, 1490, 1390, 1320, 1160	2.26(s,3H),3.05(s,3H),3.19(s,3H),4.22(s,2H),6.94-7.44 (m, 8H), 8.26 (s, 1H)
3f	346 (M^+ , 9), 226 (100), 72 (30)	3141, 1623, 1511, 1479, 1405, 1342	1.19 (t, $J=7.6$ Hz, 3H),2.59(q, $J=7.6$ Hz,2H),3.05(s,3H), 3.19 (s, 3H), 4.23 (s, 2H), 7.13-7.44(m,8H),8.21(s,1H)
5b	287 (M^+ , 100), 259 (13), 194 (40)	1710, 1510, 1475, 1350, 1280, 1190	2.40 (s, 3H), 4.27 (s, 2H), 7.15-7.98 (m, 8H)
5c	303 (M^+ , 71), 210 (15), 122 (100)	1720, 1610, 1510, 1360, 1300, 1260	3.83 (s, 3H), 4.26 (s, 2H), 6.9-7.98 (m, 8)
5d	287 (M^+ , 82), 223 (61), 194 (100)	1710, 1355, 1275, 1180, 1100, 1070	2.10(s,3H),4.23(d, $J=17.6$ Hz,1H),4.35(d, $J=17.6$ Hz,1H) , 7.23-7.98 (m, 8H)
5e	287 (M^+ , 100), 194 (51), 137 (22)	1721, 1340, 1282, 1178, 1116, 1070	2.38 (s, 3H), 4.27 (s, 2H), 7.08-7.98 (m, 8H)
5f	301 (M^+ , 100), 258 (34), 120 (56)	1716, 1509, 1352, 1278, 1182, 1110,	1.27 (t, $J=7.6$ Hz, 3H),2.70(q, $J=7.6$ Hz,2H),2.27(s,2H), 7.18-7.98 (m, 8H)

filtration and recrystallized from MeOH or MeOH-water to afford **3**.

N,N-Dimethyl-2-[2-*N*-(3-methylphenyl)sulfamoyl]phenylacetamide (**3e**) and *N,N,N',N'*-Tetramethyl-2-[2-*N*-(3-methylphenyl)sulfamoyl]phenylpropanediamide (**4**)

The precipitates obtained from **1e** according to the general procedure were mixed with hot benzene and the insoluble material was collected by filtration and recrystallized from MeOH to give **4** (24%) as white prisms: mp 226-228 °C (MeOH); IR (KBr): 3134, 1661, 1631, 1481, 1404, 1330, 1158 cm^{-1} ; MS: m/z 403 (M^+ , 10), 358 (3), 297 (11), 252 (6), 72 (100); $^1\text{H-NMR}$ (CDCl_3): δ 3.32 (s, 3H), 3.07 (s, 6H), 3.19 (s, 6H), 6.38 (s, 1H), 6.95-8.12 (m, 8H). *Anal.* Calcd for $\text{C}_{20}\text{H}_{25}\text{N}_3\text{O}_4\text{S}$: C, 59.53; H, 6.24; N, 10.41. Found: C, 59.43; H, 6.30; N, 10.28. The solvent of the filtrate was removed *in vacuo*, and the residue was recrystallized from MeOH-water to give **3e**.

2-Aryl-3,4-dihydro-2H-1,2-benzo[e]thiazin-3-one 1,1-dioxides (5). General procedure.

A mixture of **3** (1.0 mmol) in acetic acid (2.5 mL) was heated at reflux for 23-34 h. The cooled mixture was poured

Table 3. Physical Properties of Compounds (7 and 8)

Compound	Yield (%)	mp (°C)	Calcd			Found		
			C	H	N	C	H	N
7a	16 (A ^a)	196-198	63.98	5.37	9.33	63.70	5.40	9.30
7b	15 (B ^b)	196-198	64.94	5.77	8.91	64.41	5.85	9.07
7c	32 (B)	208-209	61.80	5.49	8.48	61.34	5.52	8.58
7d	22 (A)	170-172	64.94	5.77	8.91	64.89	5.80	8.94
7e	25 (B)	155-157	64.94	5.77	8.91	64.71	5.87	9.16
7f	27 (B)	190-192	65.83	6.14	8.53	65.72	6.18	8.50
8b	27 (B)	162-163	58.59	5.78	8.04	58.32	5.80	7.97
8f	15 (B)	115-117	59.64	6.12	7.73	59.29	6.09	7.83

^a Method A: n-BuLi-TMEDA was used as the base.

^b Method B: Lithium 2,2,5,5-tetramethylpiperidinate was used as the base.

into water (50 mL) and the pH of the aqueous mixture was adjusted to 1. The mixture was stirred until no longer the precipitates were formed. The precipitates were collected by filtration and recrystallized from MeOH or MeOH-water to give **5**.

2-(4-Methoxyphenyl)-3,4-dihydro-2H-1,2-benzo[e]thiazine-3-thione 1,1-dioxide (6)

A mixture of **3c** (123 mg, 0.40 mmol) and Lawesson's reagent (164 mg, 0.40 mmol) in toluene (5 mL) was heated at reflux for 6 h. After evaporation of the solvent *in vacuo*, the residue was extracted with CHCl₃, and the CHCl₃ solution was evaporated to dryness. The residue was washed with a small amount of MeOH and then recrystallized from MeOH to give **6** (84 mg, 65%) as yellow crops: mp 156-158 °C; IR (KBr): 1507, 1354, 1244, 1186, 1112 cm⁻¹; MS: *m/z* 319 (M⁺, 100), 254 (24), 240 (30), 165 (17), 139 (84), 122 (33); ¹H-NMR (CDCl₃): δ 3.84 (s, 3H), 4.74 (s, 2H), 7.00-7.95 (m, 8H). *Anal.* Calcd for C₁₅H₁₃NO₃S₂: C, 56.41; H, 4.10; N, 4.39. Found: C, 56.31; H, 4.22; N, 4.59.

2-Aryl-3-dimethylamino-2H-1,2-benzo[e]thiazine 1,1-dioxides (7) and N-Aryl-2-(N,N-dimethylthiocarbamoyl)-methylbenzenesulfonamides (8). General procedure.

Method A: To a solution of **1** (5.0 mmol) in dry THF (30 mL) cooled at -75 °C under nitrogen atmosphere was added

Table 4. Spectral Data of Compounds (7 and 8)

	MS, m/z (%)	IR (KBr)	$^1\text{H-NMR}$ (Solvent)
7a	300 (M^+ , 100), 193 39(, 166 (42)	1614, 1587, 1554, 1466, 1348, 1176	2.83(s, 3H), 5.70 (s, 1H), 7.10-7.63 (m, 9H) (CDCl_3)
7b	314 (M^+ , 100), 250 (20), 161 (47)	1606, 1585, 1549 1381, 1354, 1173	2.30 (s, 3H), 2.84 (s, 6H), 5.68 (s, 1H), 6.97-7.62 (m, 8H) (CDCl_3)
7c	330 (M^+ , 100), 251 (45), 177 (62)	1608, 1585, 1510, 1346, 1254, 1169	2.84 (s, 6H), 3.75 (s, 3H), 5.66 (s, 1H), 6.78-7.63 (m, 8H) (CDCl_3)
7d	314 (M^+ , 100), 206 (34), 159 (22)	1610, 1589, 1552, 1468, 1340, 1174	2.44 (s, 3H), 2.80 (s, 6H), 5.69 (s, 1H), 6.73-7.65 (m, 8H) (CDCl_3)
7e	314 (M^+ , 100), 235 (25), 161 (23)	1614, 1587, 1554, 1466, 1350, 1176	2.30 (s, 3H), 2.83 (s, 6H), 5.68 (s, 1H), 6.87-7.63 (m, 8H) (CDCl_3)
7f	328 (M^+ , 100), 249 (61), 175 (34)	1614, 1589, 1552, 1468, 1342, 1159	1.13 (t, $J=7.6$ Hz, 3H), 2.56 (q, $J=7.6$ Hz, 2H), 2.77 (s, 6H), 5.89 (s, 1H), 6.92-7.59 (m, 8H) (DMSO-d_6)
8b	314 (M^+ -34, 55), 178, (100), 161 (27)	3143, 1508, 1394, 1338, 1269, 1165	2.30 (s, 3H), 3.45 (s, 3H), 3.60 (s, 3H), 4.53 (s, 2H), 7.00-7.50 (m, 9H) (CDCl_3)
8f	328 (M^+ -34, 73), 249 (45), 178 (100)	3134, 1512, 1402, 1340, 1269, 1165	1.20 (t, $J=7.5$ Hz, 3H), 2.60 (q, $J=7.5$ Hz, 3H), 3.45 (s, 3H), 3.60 (s, 3H), 4.53 (s, 2H), 7.03-7.51 (m, 9H) (CDCl_3)

slowly a solution of butyllithium in hexane (1.52 M) (6.60 mL, 10.5 mmol) during 15 min and then TMEDA (1.57 mL, 10.5 mmol). The mixture was stirred at -30 °C for 30 min and then at -15 °C for 30 min. The mixture was again cooled to -75 °C, and was slowly added with a solution of *N,N*-dimethylthiocarbamoyl chloride (740 mg, 6.0 mmol) in dry THF (10 mL). After the mixture was warmed to rt, it was stirred for 90 h. The mixture was neutralized with 10% hydrochloric acid and extracted by ether. The organic layer was separated, dried over MgSO_4 , and concentrated to give an oily product, which was crystallized by addition of a small amount of ether or subjected to column chromatography (CH_2Cl_2 on silica gel) to give **7** and /or **8**.

Method B: To a solution of 2,2,6,6-tetramethylpiperidine (2.23 mL, 13.2 mmol) in dry THF (10 mL) cooled at -75 °C under nitrogen atmosphere was added slowly a solution of butyllithium in hexane (1.56 M) (7.05 mL, 11.0 mmol) during 10 min and the mixture was stirred for 30 min at rt. The mixture was again cooled to -75 °C and was added slowly with a solution of **1** (5.0 mmol) in dry THF (10 mL). It was stirred for 30 min at -30 °C, and then for 30 min at -15 °C. After a solution of *N,N*-dimethylthiocarbamoyl chloride (0.74 mg, 6.0 mmol) in dry THF (10 mL) was slowly added to the above mixture at -75 °C, the mixture was stirred for 2 days at rt. The mixture was neutralized with 10% hydrochloric acid and extracted with ether. The organic extract was dried over MgSO_4 , and evaporated. The oily

Table 5. Physical Properties of Compounds (10 and 11)

Compound	Yield (%)	mp (°C)	Calcd			Found		
			C	H	N	C	H	N
10b	60	144-145	64.59	5.42	5.38	64.36	5.55	5.39
10c	27	207-208	60.85	5.11	5.07	60.54	5.24	5.02
10e	33	124-126	64.59	5.42	5.38	64.50	5.64	5.48
10f	48	208-210	65.67	5.88	5.11	65.39	5.93	5.06
10g	16	179-181	56.58	6.65	6.60	56.40	6.72	6.80
10h ^a	5	166-167	55.40	6.74	6.46	55.93	6.86	6.57
10i	12	163-166	58.38	7.13	6.19	58.08	7.14	6.19
10j ^b	21	170-173	57.23	7.22	6.07	57.33	7.09	6.34
10k ^c	5	223-225	57.24	7.21	6.07	57.53	6.98	6.29
10l	28	144-145	64.59	5.42	5.38	64.47	5.51	5.14
11d	30	159-160	64.84	5.05	5.40	64.67	5.05	5.42
11f	6	130-132	65.91	5.53	5.12	65.90	5.60	5.13
11h	25	91-92	56.85	6.20	6.63	56.83	6.26	6.73
11k	33	104-105	58.64	6.71	6.22	58.48	6.79	6.23

^a Calcd for C₂₀H₂₈N₂O₄S₂·1/2H₂O. ^b Calcd for C₂₂H₃₂N₂O₄S₂·1/2H₂O. ^c Calcd for C₂₂H₃₂N₂O₄S₂·1/2H₂O.

residue was solidified by adding a small amount of ether and recrystallized or subjected to column chromatography (silica gel-CHCl₃) to give **7** and/or **8**.

2,2'-Ethylene-bis(*N*-substituted benzenesulfonamide)s (10) and 2-Substituted 1,2-Benzothiazole 1,1-dioxides (11).

General Procedure.

The preparative method was basically the same as that of *method A* as described above. After the reaction mixture was neutralized with 10% hydrochloric acid, the mixture was extracted with ether or diluted with water to give precipitates, and then the products were separated by recrystallization or column chromatography.

Table 6. Spectral Data of Compounds (10 and 11)

	MS, m/z (%)	IR (KBr)	$^1\text{H-NMR}$ (Solvent),
10b	520(M^+ , 25), 179 (18), 107(100)	3240, 1512, 1335, 1319, 1302, 1149	2.54 (s, 6H), 3.30 (s, 4H), 6.94-7.84 (m, 16H, ArH), 10.27 (br s, NH) (DMSO- d_6)
10c a	552 (M^+ , 18), 179 (7), 123 (100)	3276, 1511, 1391, 1332, 1254, 1160	3.21 (s, 4H), 3.64 (s, 6H), 6.78 (d, $J=9.2$ Hz, 4H), 6.97 (d, $J=9.2$ Hz, 4H), 7.33-7.79(m, 8H), 10.03(s)(DMSO- d_6)
10e	520 (M^+ , 23), 179 (30), 107 (100)	3238, 2760, 1610, 1485, 1321, 1146	2.17 (s, 6H), 3.27 (s, 4H), 6.80-7.89 (m, 16H), 10.38(s) (DMSO- d_6)
10f	548 (M^+ , 24), 179 (19), 121 (100)	3315, 1512, 1458, 1396, 1323, 1151	1.06 (t, $J=7.6$ Hz, 6H), 2.48(q, $J=7.6$ Hz, 4H), 3.24(s, 4H), 6.98 (d, $J=8.4$ Hz, 4H), 7.04(d, $J=8.4$ Hz, 4H), 7.36-7.86(m, 8H), 10.30 (s) (DMSO- d_6)
10g	424 (M^+ , 35), 245 (57), 179 (100)	3278, 2966, 1317, 1157, 1103, 1082	0.85(t, $J=7.2$ Hz, 6H), 1.45-1.54(m, 4H), 2.88-2.93(m, 4H), 3.36 (s, 4H), 5.90 (t, $J=6.0$ Hz, 2H), 7.35-8.06 (m, 8H) (CDCl_3)
10h	424 (M^+ , 26), 245 (43), 179 (100)	3257, 2976, 1471, 1325, 1302, 1161	1.08(d, $J=6.4$ Hz, 12H), 3.38(s, 4H), 3.41-3.48(m, 4H), 6.02 (d, $J=7.2$ Hz, 2H), 7.35-8.11 (m, 8H) (CDCl_3)
10i	452 (M^+ , 39), 245 (63), 179 (100)	3275, 2958, 1466 1325, 1159, 1063	0.82(t, $J=7.2$ Hz, 6H), 1.23-1.32(m, 4H), 1.41-1.49(m, 4H), 2.90-3.36(m, 4H), 3.36(s, 4H), 5.88(t, $J=5.8$ Hz, 2H), 7.36-8.06 (m, 8H) (CDCl_3)
10j	452 (M^+ , 22), 245 (100), 179 (36)	3280, 2956, 1469, 1323, 1155, 1078	0.86(d, $J=6.8$ Hz, 12H), 1.68-1.78(m, 2H), 2.74(t, $J=6.4$ Hz, 2H), 6.02 (t, $J=6.0$ Hz, 2H), 7.36-8.06 (m, 8H) (CDCl_3)
10k	438 (M^+ -14, 12), 382 (68), 179 (100)	3248, 2968, 1473, 1392, 1306, 1142	1.19 (s, 18H), 3.40 (s, 4H), 6.44(s, 2H), 7.34-8.15(m, 8H) (CDCl_3)
10^b		3203, 1456, 1427, 1315, 1153, 1065	3.25 (s, 4H), 4.09 (d, $J=6.2$ Hz, 4H), 5.82(t, $J=6.2$ Hz, 2H), 7.09-8.02 (m, 8H) (CDCl_3)
11d	259 (M^+ , 65), 194 (100), 118 (26)	1493, 1460, 1284, 1263, 1169, 1134	2.35 (s, 3H), 4.87 (s, 2H), 7.31-7.96 (m, 4H) (DMSO- d_6)
11f	273 (M^+ , 93), 258 (100), 180 (44)	2964, 1514, 1469, 1300, 1269, 1167	1.25 (t, $J=7.6$ Hz, 3H), 2.66 (q, $J=7.6$ Hz, 2H), 4.86 (s, 2H), 7.25-7.89 (m, 4H) (CDCl_3)
11h	211 (M^+ , 5), 196 (100), 91 (30)	2976, 1458, 1396, 1279, 1205, 1163	1.39(d, $J=6.4$ Hz, 6H), 4.06-4.16(m, 1H), 4.38(s, 2H), 7.39-7.80 (m, 4H) (CDCl_3)
11k	210 (M^+ -15, 100), 167 (15), 119 (19)	1456, 1277, 1221, 1161, 1130, 1001	1.57 (s, 9H), 4.46 (s, 2H), 7.36-7.76 (m, 4H) (CDCl_3)

^a $^{13}\text{C-NMR}$ (DMSO- d_6): 33.71 ($\underline{\text{C}}\text{H}_2$), 55.12 ($\underline{\text{O}}\underline{\text{C}}\text{H}_3$), 114.21, 122.92, 126.39, 128.99, 129.10, 131.70, 132.71, 137.60, 140.24, 156.21.

^b Satisfactory MS spectrum was not taken.

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