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Sulfur-Containing Derivatives of 1,4-Naphthoquinone, Part 2: Sulfenyl Derivative Synthesis

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ABSTRACT: *Sulfenylchlorides, sulfenates, and sulfenamides of 1,4-naphthoquinone were synthesized, and different methods of their syntheses were investigated. Synthesized sulfenates and sulfenamides are stable due to the large electron-withdrawing potential of the conjugated quinonic system. Obtained mono- and bi-functional sulfenderivatives are very important in organic synthesis of different new heterocyclic compounds.* © 2005 Wiley Periodicals, Inc. Heteroatom Chem 16:587–598, 2005; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20157

INTRODUCTION

Recently, pharmaceutics and scientists have been greatly interested in sulfenic-containing substances because they are synthones for further conversions and they are compounds with a wide spectrum of biological activity. Besides, derivatives of 1,4-naphthoquinone have been revealed as bacteriostatic, antiviral, and fungistatic active. The drugs on the base of the naphthoquinone possess a high antioxidant, anticancer, and cytostatic activity.

Compounds of the bivalent sulfur such as sulfenylchlorides and sulfenamides have a high reaction ability that conditions their wide use within organic synthesis. Due to high polarity and lability of S—Cl, S—N bonds, they easily react with both nucleophilic and electrophilic reagents, and thus may be to used for synthesis of sulfur compounds with carbon and heteroatom bonds such as sulfides, disulfides, sulfinamides, sulfenates, sulfonates, sulfochlorides, aminosulfonics, thioammonium, and sodium salts.

Bifunctional sulfenylic derivatives that contain other functional groups(Cl, Br, C=O, S—Cl, ClC=O, ClC=N, CCl_3, CF_2Cl, and others) are used in the synthesis of different heterocyclic compounds.

Further, sulfenylchlorides and sulfenamides are used in macromolecular chemistry as monomers [1], in the production of such widely known biologically active preparations such as phtalan, captan [2,3], euparen, euparen–M [4,5], as well as in the production of pharmaceutical preparations [3,8], and vulcanizing agents [9,10–14], what makes their perspective clear in the practical sense.

Our purpose is the synthesis of compounds, which combine a naphthoquinone and sulfenylic fragments that would expand the spectrum of biological action.

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RESULTS AND DISCUSSION

We have investigated some methods of synthesis of the new sulfenylic derivatives of 1,4-naphthoquinone, which are not described in the literature before.

Synthesis of the Sulfenylchlorides of 1,4-Naphthoquinone

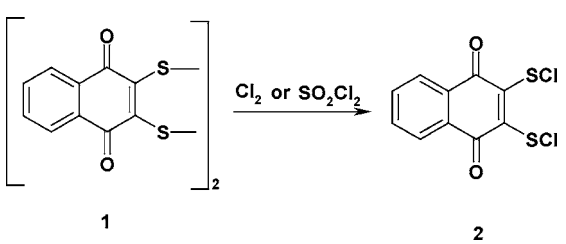
In the literature, various methods of the synthesis of sulfenylchlordies [15–17] are described. The main methods consist in chlorinolysis of disulfides, thiols, and sulfides. For activation of S—S, S—H, and C—S bonds and to carry out chlorinolysis in many cases, it is necessary to select a catalyst, which would be able to increase electrophilic properties of the bonds in combination with an effective chlorinating agent.

It is known [18] that sulfuryl chloride in chlorinolysis of disulphides is more effective than chlorine. Excess amount of chlorine leads to the formation of polychlorides $RSCl_2SCl_2R$ and $RSCl_3$ [19], which are unstable and pollute sulfenylchlorides. The reaction is carried out in anhydrous chloroform or carbon tetrachloride.

We have investigated the chlorinolysis of thiols, disulfides, and sulfides of 1,4-naphthoquinone under the action of the some chlorinating agents—chlorine, sulfuryl chloride, and *N*-chlorosuccinimide with and without catalytic conditions.

Chlorinolysis of sulfur-containing derivatives of 1,4-naphthoquinone by chlorine and sulfuryl chloride was carried out with different ratios of reagents. In the case of thiols, 1:1.4 was found to be the best ratio. When a smaller amount of chlorine is used, poor yields of sulfenylchloride are obtained, and a larger portion of disulfide is formed.

Chlorinolysis of disulfides and sulfides does not work at room temperature. Under long time boiling with excess amount of chlorine or sulfuryl chloride, the decomposition of disulfide and sulfide bonds and chlorination of aromatic ring and in a side chain were observed. One exception was 2,3-disulfenylchloride-1,4-naphthoquinone (**2**), obtained from *bis*-disulfide **1** [20] (Scheme 1).



1 2

SCHEME 1

Chlorination of sulfides **3a–h, 6, 9** led to reaction mixtures of sulfenylchlorides **4a–h, 7,** respectively and by-products **5a–h, 8, 2** which are difficult to separate by chromatography on silica gel (Scheme 2).

It is known that sulfuryl chloride slowly reacts with aromatic disulfides. In the case of electron-withdrawing groups in *ortho* to *para* position, the reaction does not take place.

Parfyonov and Fomin [21] established that triphenylphosphine carbamide, tertiary amines, and amides of carbonic acids are effective catalysts of the chlorinolysis of aromatic disulfides. Authors have suggested a mechanism of the catalysis.

Various attempts to use these methods for disulfides, thiols, and sulfides at room temperature have failed. At the boiling of reaction mixture, the chlorination of alkyl groups and aromatic system and the elimination of aminosubstitutes were also observed. It decreased yields of sulfenylchlorides to 20–30%. The synthesis of 2,3-disulfenylchloride-1,4-naphthoquinone takes longer, than in the case of chlorination of *bis*-disulfide by chlorine.

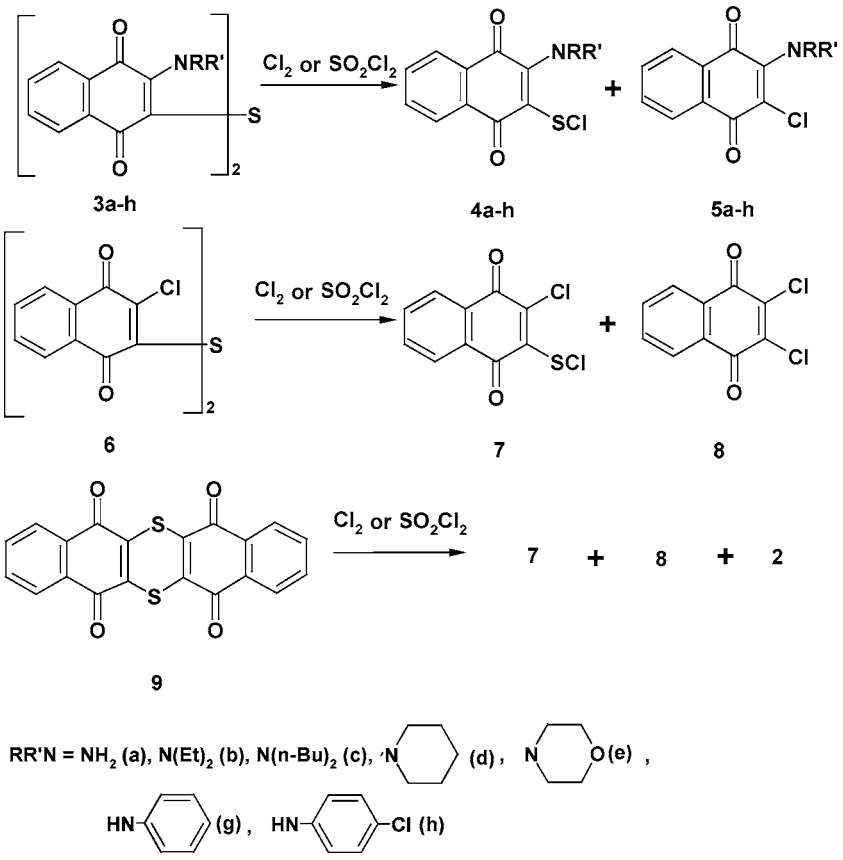
N-Chlorosuccinimide was used as a chlorinating agent to avoid such side effects. The reaction is easily completed under room temperature without forming by-products, and succinimide is easy to separate from a reaction mixture.

In order to obtain sulfenylchlorides **4a–h, 7**, we used amino-, chloro-, thiols **10a–h, 12**; synthesis of these compounds has been realized in the two different ways [20]. The reaction was carried out similarly, as described in [22,23], in inert solvents (Scheme 3). The formation of the products was observed after 25–35 min: sulfenylchloride dissolves, and succinimide precipitates.

Therefore, sulfenylchlorides are better synthesized by the interaction of thiols with *N*-chlorosuccinimide or chlorine in inert solvents at room temperature. Yields of obtained sulfenylchlorides are 59–75%.

Synthesis of Sulfenates of 1,4-Naphthoquinone

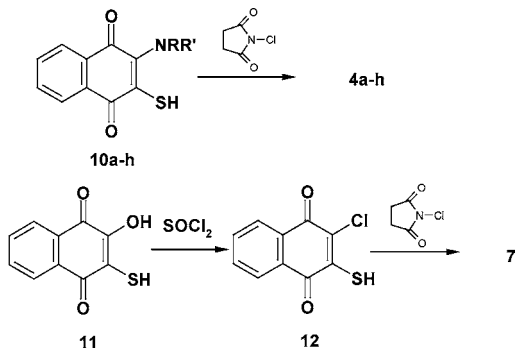
It is known that esters of sulfenic acids have been obtained by the reaction of sulfenylchlorides with alcoholate (phenolate) or alcohol (phenol) in the presence of the base [15,24–26]. The reaction is not always facile. Obtained sulfenates can react with sulfenylchloride-forming disulfides and other by-products due to high nucleophility of sulfur. The reaction of sulfenylchlorides with a strong electron-withdrawing group at the sulfur atom is more facile. This reduces the nucleophility of the sulfur atom.



SCHEME 2

We carried out synthesis of sulfenates **13–19a–d, 20a–d, 21a–d** by the interaction of sulfenylchlorides **4a–h, 7, 2** with aliphatic alcohol and phenols in dry inert solvents in the presence of triethylamine (Scheme 4).

The esters of sulfenic acids are labile compounds [15, 24–26]. Synthesized sulfenates of 1,4-naphthoquinone are stable substances. This is explained by the influence of a conjugated quinonic system with a large electron-withdrawing potential.



RR'N as in Scheme 2.

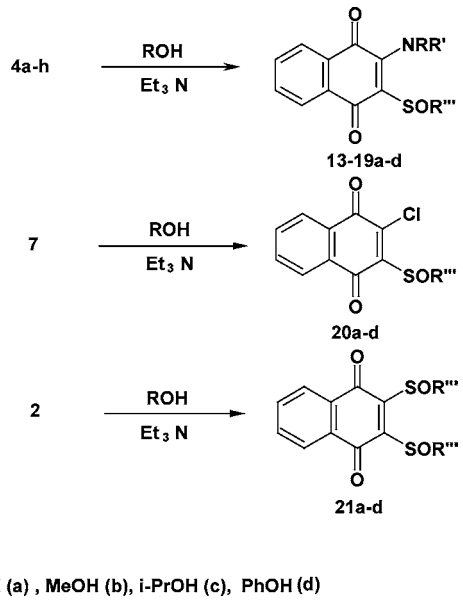
SCHEME 3

Synthesis of the Sulfenamides of the 1,4-Naphthoquinone

We carried out the synthesis of the sulfenamides **22a–c, 23–29a–c, 30a–c** by the interaction of obtained sulfenylchlorides with the primary, secondary, alkyl, and aryl amines in the presence of triethylamine (Scheme 5). Such a reaction occurred with high yields and without formation of by-products quickly under mild conditions.

Sulfenamides have been obtained by an alternative synthesis, which form disulfides [20] and primary, secondary alkyl, and aryl amines in the presence of silver nitrate similar to [27]. Sulfenamide dissolves, and silver mercaptide precipitates (Scheme 6).

This method is less acceptable because initial disulfides are difficult to purify, and the reaction is carried out under harder conditions in protonic solvents. This reduces yields and purity of

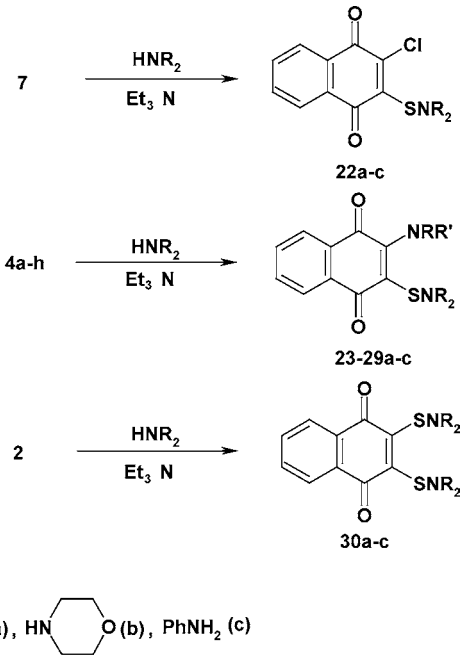


RR'N as in Scheme 2.

SCHEME 4

sulfenamides. The structures of the obtained compounds were confirmed by NMR, IR, and the element analysis.

Prepared mono- and bi-functional sulfenyl derivatives are useful for the synthesis of the heterocyclic compounds with different functional groups.



RR'N as in Scheme 2.

SCHEME 5

EXPERIMENTAL

Melting points were measured on a Nagema melting point apparatus and are uncorrected. ^{1}H NMR spectra were recorded on Varian VXR (300 MHz) spectrometer as solutions in DMSO-d_6 with as an internal standard. IR spectra were recorded on Specord M80 in the tablets of KBr. Materials used were disulfides, thiols, and sulfides [20].

General Procedure of Sulfenylchloride-1,4-naphthoquinones Synthesis **4a–h, 7** *from Thiols* **10a–h, 12**

Method A. 0.05 mol of thiol and 0.05 mol of *N*-chlorosuccinimide were suspended in carbon tetrachloride. The reaction mixture was left at room temperature for 24 h and filtered. The precipitate was washed by small amount of carbon tetrachloride. The filtrate was evaporated. Residue was recrystallized from benzene.

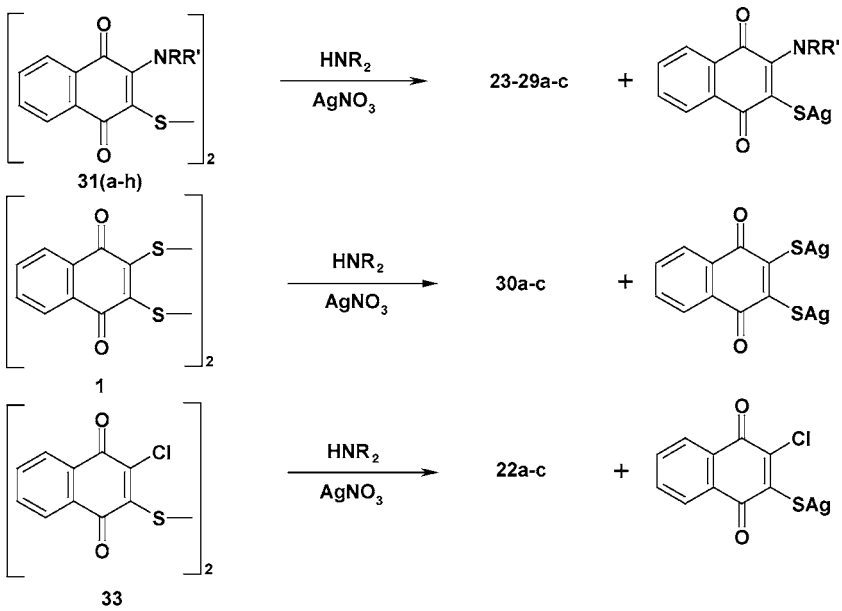
Method B. 0.05 mol of thiol (or **2**) was suspended in carbon tetrachloride, and 0.1 mol of cl_2(solution in CCl_4) was added. The reaction mixture was left at room temperature for 24 h and filtered. The precipitate was washed by small amount of carbon tetrachloride. The filtrate evaporated. Residue was recrystallized from benzene. 2,3-Disulfenylchloride-1,4-naphthoquinone **2** was obtained from **1**. See method B.

See Table 1 for yields and elemental analysis of sulfenylchlorides **4a–h, 7, 2** obtained from **10a–h, 12, 1**.

General Procedure of the Synthesis of Sulfenates of 1,4-Naphthoquinone **13–19a–d, 20a–d, 21a–d** *from the Sulfenylchlorides* **4a–h, 2, 7**

The mixture of alcohol or phenol (0.2 mol) and triethylamine (0.2 mol) was stirred for 10 min in benzene. Benzene solution of sulfenylchloride (0.2 mol) was added. The reaction mixture was left at room temperature for 24 h, then warmed up to 40°C for 5–6 h. The reaction mixture was evaporated. Residue was recrystallized from hexane.

See Table 2 for yields and elemental details of sulfenates **13–19a–d, 20a–d, 21a–d** obtained from sulfenchlorides **4a–h, 2, 7**.



RR' N as in Scheme 2.

R_2N as in Scheme 5.

SCHEME 6

General Procedure of Sulfenamide-1,4-naphthoquinones Synthesis **22a–c, 23–29a–c, 30a–c** *from the Sulfenylchlorides* **4a–h, 2, 7** *(A) and Disulfides* **1, 31–33a–h** *(B)*

Method A. The mixture of amine (0.025 mol) and triethylamine (0.025 mol) was stirred for 10 min in benzene. Benzene solution of sulfenylchloride (0.025 mol) was added. The reaction mixture was left at room temperature for 24 h, then was warmed up to 40°C for 5–6 h. The reaction mixture was evaporated. Residue was recrystallized from hexane.

TABLE 1 Sulfenylchlorides **4a–h, 7, 2** from **10a–h, 12, 1**

	Yield (%)	*Calculated Found (%)*				
	A/B	*C*	*H*	*S*	*Cl*	*N*
4a	66/63	56.85	4.77	10.84	11.99	4.74
		56.91	4.80	10.67	11.84	4.65
4b	59/54	61.44	6.30	9.11	10.07	3.98
		61.32	6.37	9.01	10.11	3.91
4c	68/65	58.53	4.58	10.42	11.52	4.55
		58.42	4.46	10.46	11.54	4.60
4d	72/75	54.28	3.90	10.35	11.44	4.52
		54.29	3.87	10.42	11.39	4.56
4e	67/65	60.86	3.19	10.15	11.23	4.44
		60.79	3.21	10.18	11.29	4.46
4g	73/72	54.87	2.59	9.16	20.25	4.00
		54.76	2.61	9.21	20.31	4.01
4h	62/61	52.45	2.14	9.97	15.53	4.50
		52.36	2.17	9.89	15.63	4.42
7	63/66	46.36	1.56	12.37	27.36	
		46.31	1.58	12.42	27.41	
2	62/63	41.25	1.38	22.02	24.35	
		41.31	1.34	22.01	24.41	

TABLE 2 Sulfenates **13–19a–d, 20a–d, 21a–d** from sulfenchlorides **4a–h, 2, 7**

	Yield (%)	*Calculated Found (%)*				
	A/B	*C*	*H*	*S*	*N*	*Cl*
13a	70	61.83	5.88	11.00	4.81	
		61.85	5.94	11.02	4.75	
13b	73	62.93	6.27	10.50	4.59	
		62.98	6.31	10.47	4.61	
13c	72	63.92	6.63	10.04	4.38	
		63.96	6.72	10.07	4.36	
13d	71	67.97	5.42	9.07	3.96	
		68.01	5.46	9.09	3.93	
14a	73	64.84	6.95	9.62	4.20	
		64.87	6.98	9.64	4.26	
14b	69	65.68	7.25	9.23	4.03	
		65.70	7.28	9.21	4.01	
14c	72	66.45	7.53	8.87	3.87	
		66.49	7.49	8.82	3.91	
14d	74	69.85	6.37	8.11	3.54	
		69.81	6.41	8.13	3.49	
15a	68	63.35	5.65	10.57	4.62	
		63.40	5.60	10.58	4.64	
15b	72	64.33	6.03	10.10	4.41	
		64.37	6.07	10.07	4.38	
15c	74	65.23	6.39	9.67	4.23	
		65.19	6.37	9.62	4.21	
15d	73	69.02	5.24	8.77	3.83	
		69.04	5.27	8.72	3.85	
16a	76	59.00	4.95	10.50	4.59	
		59.03	4.91	10.47	4.60	
16b	71	60.17	5.37	10.04	4.39	
		60.23	5.35	10.05	4.41	
16c	74	61.24	5.74	9.62	4.20	
		61.20	5.78	9.64	4.23	
16d	69	65.38	4.66	8.73	3.81	
		65.40	4.61	8.68	3.82	
17a	73	65.53	4.21	10.30	4.60	
		65.51	4.27	10.34	4.58	
17b	76	66.44	4.65	9.85	4.30	
		66.42	4.67	9.89	4.35	
17c	72	67.24	5.05	9.45	4.13	
		67.32	5.00	9.50	4.10	
17d	78	70.76	4.05	8.59	3.75	
		70.71	4.01	8.63	3.78	
18a	67	59.05	3.05	9.27	4.05	
		59.00	3.08	9.31	4.01	
18b	69	60.08	3.92	8.91	3.89	
		60.11	3.95	8.93	3.85	
18c	70	61.04	4.31	8.58	3.75	
		61.06	4.35	8.62	3.78	
18d	68	64.79	3.46	7.86	3.43	
		64.82	3.52	7.91	3.41	
19a	71	51.88	2.77	12.59	4.05	13.92
		51.91	2.79	12.61	4.10	13.90
19b	72	53.64	3.38	11.93	3.89	13.19
		53.61	3.39	11.96	3.91	13.15
19c	69	55.22	3.92	11.34	3.75	12.54
		55.23	3.95	11.36	3.69	12.62
19d	71	60.67	2.86	10.12	3.43	11.19
		60.69	2.90	10.14	3.45	11.21
20a	73	51.88	2.77	12.59		13.92
		51.83	2.76	12.61		13.96

(Continued)

TABLE 2 Continued

	Yield (%) A/B	Calculated Found (%) C	H	S	N	Cl
20b	72	53.64	3.38	11.93		13.19
		53.67	3.42	11.91		13.14
20c	76	55.22	3.92	11.34		12.54
		55.25	3.96	11.36		12.49
20d	75	60.67	2.86	10.12		11.19
		60.73	2.90	10.15		11.21
21a	74	51.05	3.57	22.71		
		51.09	3.62	22.76		
21b	70	54.18	4.55	20.66		
		54.22	4.59	20.68		
21c	76	56.78	5.36	18.95		
		56.81	5.43	18.93		
21d	73	65.01	3.47	15.78		
		65.06	3.50	15.84		

Method B. 0.027 mol of silver nitrate was dissolved in 100 mL of methanol. The solution was cooled in an ice bath; an equivalent amount of disulfide was added. The reaction mixture was left at constant stirring for 15 min. Then 0.027 mol of amine was added, and mixture was stirred overnight. Precipitated silver mercaptide was removed by filtration; solvent was removed to give a residue which was recrystallized from hexane.

See Table 3 for yields and elemental details of sulfenamides **22a–c, 23–29a–c, 30a–c** from sulfenylchlorides **4a–h, 2, 7** (A) and disulfides **1, 31–33a–h** (B) and Table 4 for NMR, IR data of the synthesized compounds.

TABLE 3 Sulfenamides **22a–c, 23–29a–c, 30a–c** from Sulfenylchlorides **4a–h, 2, 7** (A) and Disulfides **1, 31-33a-h** (B)

	Yield (%) A/B	Calculated Found (%) C	H	S	N	Cl
23a	67/54	62.48	5.59	11.12	9.71	
		62.47	5.67	11.10	9.75	
23b	64/52	57.92	4.86	11.04	9.65	
		57.89	4.83	11.07	9.67	
23c	69/56	64.85	4.08	10.82	9.45	
		64.81	4.11	10.85	9.43	
24a	60/52	66.25	7.02	9.31	8.13	
		66.18	7.06	9.38	8.15	
24b	67/52	62.40	6.40	9.25	8.09	
		62.43	6.38	9.27	8.05	
24c	71/60	68.16	5.72	9.10	7.95	
		68.14	5.68	9.13	7.93	
25a	72/59	68.96	8.05	8.00	6.99	
		68.93	8.01	8.05	6.95	
25b	70/56	65.64	7.51	7.97	6.96	
		65.61	7.60	7.91	7.01	
25c	75/63	70.56	6.91	7.85	6.86	
		70.62	6.87	7.84	6.91	
26a	76/62	67.37	6.79	8.99	7.86	
		67.41	6.74	8.97	7.95	
26b	74/63	63.66	6.19	8.94	7.81	
		63.62	6.21	8.91	7.91	
26c	73/59	69.21	5.53	8.80	7.69	
		69.19	5.49	8.85	7.76	

(Continued)

TABLE 3 Continued

	Yield (%)	*Calculated Found (%)*				
	A/B	*C*	*H*	*S*	*N*	*Cl*
27a	71/63	63.66	6.19	8.94	7.81	
		63.78	6.08	8.91	7.86	
27b	72/59	59.98	5.59	8.90	7.71	
		59.89	5.56	8.95	7.81	
27c	70/61	65.56	4.95	8.75	7.64	
		65.62	4.91	8.79	7.58	
28a	73/65	69.21	5.53	8.80	7.69	
		69.25	5.62	8.82	7.62	
28b	74/60	65.56	4.95	8.75	7.64	
		65.52	4.97	8.71	7.69	
28c	69/58	70.95	4.33	8.61	7.52	
		70.91	4.38	8.67	7.43	
29a	65/61	63.23	4.80	8.04	7.02	8.89
		63.19	4.83	8.09	6.92	8.83
29b	72/69	59.92	4.27	8.00	6.99	8.84
		59.89	4.24	8.10	6.92	8.91
29c	71/65	64.94	3.72	7.88	6.88	8.71
		64.92	3.78	7.92	6.85	8.69
22a	74/67	58.53	4.58	10.42	4.55	11.52
		58.48	4.48	10.36	4.52	11.57
22b	69/61	54.28	3.90	10.35	4.52	11.44
		54.32	3.79	10.22	4.63	11.48
22c	73/66	60.86	3.19	10.15	4.44	11.23
		60.92	3.25	10.07	4.47	11.19
30a	71/68	61.82	6.23	16.50	7.21	
		61.95	6.18	16.56	7.28	
30b	73/64	55.08	5.14	16.34	7.14	
		55.12	5.24	16.48	7.10	
30c	72/65	65.32	3.99	15.85	6.93	
		65.24	4.01	15.77	6.96	

TABLE 4 NMR, IR Data of the Synthesized Compounds

	Formula, mp (°C)	*^1H (δ, ppm)*	*IR (cm^{-1})*
4a[a]	$C_{10}H_6NSClO_2$ 116	7.63–8.29 (6H, m, ArH and NH_2)	1655 (C=O), 1358 (*tert*-N)
4b[a]	$C_{14}H_{14}NSClO_2$ 124	1.08 (12H, t, CH_3), 3.41 (8H, q, CH_2), 7.66; 7.56 (4H, td, CH_{Ar}), 8.12; 8.05 (4H, dd, CH_{Ar})	1650 (C=O), 1350 (*tert*-N)
4c[a]	$C_{18}H_{22}NSClO_2$ 156	0.84 (12H, t, $^3J_{HH} = 9.3$ Hz, CH_3), 1.32 (16H, m, CH_2), 3.06 (8H, m, CH_2), 8.03; 8.04 (4H, td, CH_{Ar}), 8.12; 8.17 (4H, dd, CH_{Ar})	1655 (C=O), 1360 (*tert*-N)
4d[a]	$C_{14}H_{18}NSClO_2$ 117	1.31 (12H, m, CH_2), 3.71 (8H, m, CH_2), 7.46; 7.78 (4H, td, CH_{Ar}), 7.98; 8.17 (4H, dd, CH_{Ar})	1620, 1656 (C=O), 1340 (*tert*-N)
4e[a]	$C_{14}H_{12}NSClO_3$ 134	δ 3.49 (16H, m, $CH_{2morpholine}$), 7.56; 7.67 (4H, td, CH_{Ar}), 8.02; 8.05 (4H, dd, CH_{Ar})	1612, 1648 (C=O), 1355 (*tert*-N)
4g[a]	$C_{16}H_{10}NSClO_2$ 162	δ 7.05 (10H, m, Ar), 7.33 (2H, s broad, NH), 7.62; 7.82 (4H, td, CH_{Ar}), 7.66; 8.05 (4H, dd, CH_{Ar})	3160 (–NH–), 1610, 1650 (C=O)
4h[a]	$C_{16}H_9NSCl_2O_2$ 145	6.94 (4H, d, $^3J_{HH} = 8.4$ Hz, CH_{Ar}), 7.1 (4H, d, $^3J_{HH} = 8.4$ Hz, CH_{Ar}), 7.41 (2H, s broad, NH), 7.63; 7.70 (4H, td, CH_{Ar}), 8.16; 8.19 (4H, dd, CH_{Ar})	3160 (–NH–), 1612, 1648 (C=O)
7[a]	$C_{10}H_4SCl_2O_2$ 105	δ 7.79 (2H, m, CH_{Ar}), 8.08; 8.16 (2H, dd, CH_{Ar}), 8.51 (1H, s, SH)	1650 (C=O)
2[a]	$C_{10}H_4S_2O_2Cl_2$ 195	7.65 (4H, tm, CH_{Ar}), 8.22 (4H, dd, CH_{Ar})	1650 (C=O)

(Continued)

TABLE 4 Continued

	Formula, mp (°C)	*^1H (δ, ppm)*	*IR (cm^{-1})*
13a[a]	$C_{11}H_9NSO_3$ 86	7.63; 7.75 (2H, td, CH_{Ar}), 7.98; 8.01 (2H, dd, CH_{Ar}); 6.7 (2H, s, NH_2); 2.7 (3H, s, –OCH_3)	2813 (OCH_3),1655 (C=O), 1358 (*tert*-N), 1590 (NH_2)
13b[a]	$C_{12}H_{11}NSO_3$ 92	7.66; 7.56 (4H, td, CH_{Ar}), 8.12; 8.05 (4H, dd, CH_{Ar}); 6.73 (2H, s, NH_2); 3.97 (2H, q, CH_2); 1.1 (3H, s, CH_3)	1640 (C=O),1560 (NH_2), 1370 (CH_3)
13c[a]	$C_{13}H_{13}NSO_3$ 87	7.45; 7.79 (4H, td, CH_{Ar}), 7. 91; 8.15 (4H, dd, CH_{Ar}); 0.80–0.85 (6H, dt, $2CH_3$); 2.14–2.30 (1H, m, CH); 3.50–3.53 (2H, m, –OCH_2); 6,71 (2H, s, NH_2)	1648 (C=O), 1610 (NH_2), 1370 ($C(CH_3)_2$)
13d[a]	$C_{16}H_{12}NSO_3$ 121	7.71; 7.65 (4H, td, CH_{Ar}), 8.07; 8.01 (4H, dd, CH_{Ar}); 6.69 (2H, s, NH_2); 6,89–7.12 (5H, m, CH_{Ar})	1652 (C=O), 1590 (NH_2), 1580 (Ar)
14a[a]	$C_{15}H_{17}NSO_3$ 91	7.56; 7.67 (4H, td, CH_{Ar}), 8.17; 8.10 (4H, dd, CH_{Ar}); 3.44–3.49 (4H, m, –$N(CH_2)_2$); 2.78 (3H, s, –OCH_3); 1.13 (6H, t, $2CH_3$)	2817 (OCH_3), 1648 (C=O)
14b[a]	$C_{16}H_{19}NSO_3$ 98	7.72; 7.63 (4H, td, CH_{Ar}), 8.12; 8.04 (4H, dd, CH_{Ar}); 3.98 (2H, m, –OCH_2); 3.44–3.49 (4H, m, –$N(CH_2)_2$); 3.96 (2H, q, –OCH_2)	1665 (C=O), 1380 (CH_3)
14c[a]	$C_{17}H_{21}NSO_3$ 113	7.66; 7.56 (4H, td, CH_{Ar}), 8.08; 8.01 (4H, dd, CH_{Ar}); 3.56–3.57 (2H, m, –OCH_2); 3.44–3.49 (4H, m, –$N(CH_2)_2$); 2.23–2.35 (1H, m, CH); 1.08 (6H, t, $2CH_3$); 0.78–0.81 (6H, dt, $2CH_3$)	1648 (C=O), 1354 ($C(CH_3)_2$)
14d[a]	$C_{20}H_{19}NSO_3$ 110	7.78; 7.64 (4H, td, CH_{Ar}), 8.18; 8.11 (4H, dd, CH_{Ar}); 7.01–7.26 (5H, m, CH_{Ar}); 3.6 (4H, q, –$N(CH_2)_2$); 1.13 (6H, t, $2CH_3$)	1650 (C=O), 1500 (Ar)
15a[a]	$C_{19}H_{25}NSO_3$ 78	7.65; 7.57 (4H, td, CH_{Ar}), 8.14; 8.07 (4H, dd, CH_{Ar}); 3.03–3.21 (4H, m, $2CH_2$); 2.75 (3H, s, –OCH_3); 1.23–1.41 (4H, m, $2CH_2$); 0.8–0.98 (6H, t, $2CH_3$)	2810 (OCH_3), 1660 (C=O), 1380 (CH_3)
15b[a]	$C_{20}H_{27}NSO_3$ 92	7.81; 7.69 (4H, td, CH_{Ar}), 8.15; 8.08 (4H, dd, CH_{Ar}); 3.95–4.00 (2H, m, –OCH_2); 3.08–3.13 (4H, m, –$N(CH_2)_2$); 1.23–1.39 (8H, m, $4CH_2$); 1.10 (3H, t, CH_3); 0.86–0.91 (6H, t, $2CH_3$)	1610 (C=O), 1375 (CH_3)
15c[a]	$C_{21}H_{29}NSO_3$ 98	7.70; 7.61 (4H, td, CH_{Ar}), 8.10; 8.02 (4H, dd, CH_{Ar}); 3.57–3.61 (2H, m, –OCH_2); 3.09–3.12 (4H, m, –$N(CH_2)_2$); 2.23–2.35 (1H, m, CH); 1.24–1.39 (8H, m, $4CH_2$); 0.82–0.90 (6H, t, $2CH_3$)	1650 (C=O), 1470 (CH_3), 1385 ($C(CH_3)_2$)
15d[a]	$C_{24}H_{27}NSO_3$ 101	7.45; 7.56 (4H, td, CH_{Ar}), 8.22; 8.12 (4H, dd, CH_{Ar}); 7.03–7.35 (5H, m, CH_{Ar}); 3.1–3.14 (4H, m, –$N(CH_2)_2$); 1.27–1.42 (8H, m, $4CH_2$); 0.84–0.98 (6H, m, $2CH_3$)	1650 (C=O), 1575 (Ar), 1375 (CH_3)
16a[a]	$C_{16}H_{17}NSO_3$ 119	7.71; 7.64 (4H, td, CH_{Ar}), 8.20; 8.16 (4H, dd, CH_{Ar}); 3.65–3.85 (4H, m, –$N(CH_2)_2$); 2.76 (3H, s, –OCH_3); 1.24–1.54 (6H, m, $2CH_3$)	2810 (OCH_3), 1660 (C=O), 1362 (*tert*-N)
16b[a]	$C_{17}H_{19}NSO_3$ 118	7.63; 7.52 (4H, td, CH_{Ar}), 8.12; 8.02 (4H, dd, CH_{Ar}); 3.69–3.83 (4H, m, –$N(CH_2)_2$); 4.01 (2H, m, –OCH_2); 1.27–1.41 (6H, m, $2CH_3$); 1.19 (3H, t, CH_3)	1661 (C=O), 1470 (CH_3), 1345 (*tert*-N)
16c[a]	$C_{18}H_{21}NSO_3$ 130	7.56; 7.47 (4H, td, CH_{Ar}), 8.02; 8.05 (4H, dd, CH_{Ar}); 3.68–3.84 (4H, m, –$N(CH_2)_2$); 3.55–3.57 (2H, m, –OCH_2); 2.28–2.42 (1H, m, CH); 1.31–1.48 (6H, m, $2CH_3$); 0.87–0.89 (6H, dt, $2CH_3$)	1645 (C=O), 1380 ($C(CH_3)_2$), 1342 (*tert*-N)
16d[a]	$C_{21}H_{19}NSO_3$ 125	7.66; 7.58 (4H, td, CH_{Ar}), 7.98; 8.01 (4H, dd, CH_{Ar}); 7.06–7.42 (5H, m, CH_{Ar}); 3.73–3.86 (4H, m, –$N(CH_2)_2$); 1.27–1.43 (6H, m, $2CH_3$)	1655 (C=O), 1570 (Ar), 1352 (*tert*-N)
17a[a]	$C_{15}H_{15}NSO_4$ 142	7.66; 7.56 (4H, td, CH_{Ar}), 8.12; 8.05 (4H, dd, CH_{Ar}); 3.63–3.71 (4H, m, –$N(CH_2)_2$); 3.33–3.51 (4H, m, –$O(CH_2)_2$); 2.76 (3H, s, –OCH_3)	2819 (OCH_3), 1649 (C=O), 1340 (*tert*-N),
17b[a]	$C_{16}H_{17}NSO_4$ 131	7.65; 7.79 (4H, td, CH_{Ar}), 8.12; 8.05 (4H, dd, CH_{Ar}); 3.96 (2H, q, –OCH_2); 3.61–3.72 (4H, m, –$N(CH_2)_2$); 3.38–3.51 (4H, m, –$O(CH_2)_2$); 1.15 (3H, t, CH_3)	1661 (C=O), 1450 (CH_3), 1348 (*tert*-N)
17c[a]	$C_{17}H_{19}NSO_4$ 120	7.71; 7.62 (4H, td, CH_{Ar}), 7.98; 8.01 (4H, dd, CH_{Ar}); 3.55–3.61 (2H, m, CH_2); 3.51–3.55 (4H, m, –$N(CH_2)_2$); 3.38–3.47 (4H, m, –$O(CH_2)_2$); 2.27–2.35 (1H, m, CH); 0.87–0.94 (6H, dt, $2CH_3$)	1645 (C=O), 1390 ($C(CH_3)_2$), 1360 (*tert*-N)

(Continued)

TABLE 4 Continued

	Formula, mp (°C)	*^1H (δ, ppm)*	*IR (cm^{-1})*
17d[a]	$C_{20}H_{17}NSO_4$ 135	7.47; 7.68 (4H, td, CH_{Ar}), 8.10; 8.03 (4H, dd, CH_{Ar}); 7.01–7.36 (5H, m, CH_{Ar}); 3.55–3.67 (4H, m, –$N(CH_2)_2$); 3.41–3.52 (4H, m, –$O(CH_2)_2$)	1651 (C=O), 1500 (Ar), 1362 (*tert*-N)
18a[a]	$C_{17}H_{13}NSO_3$ 124	7.79; 7.81 (4H, td, CH_{Ar}), 8.15; 8.05 (4H, dd, CH_{Ar}); 7.98 (1H, s, –NH); 7.06–7.45 (5H, m, CH_{Ar}); 2.85 (3H, s, –OCH_3)	3160 (–NH–), 2820 (OCH_3), 1649 (C=O), 1600 (Ar)
18b[a]	$C_{18}H_{15}NSO_3$ 128.5	7.66; 7.56 (4H, td, CH_{Ar}), 8.12; 8.05 (4H, dd, CH_{Ar}); 7.83 (1H, s, –NH); 7.07–7.27 (5H, m, CH_{Ar}); 3.96 (2H, t, CH_2); 1.1 (3H, t, CH_3)	3158 (–NH–), 1660 (C=O), 1500 (Ar), 1470 (CH_3)
18c[a]	$C_{19}H_{17}NSO_3$ 131–132	7.60; 7.74 (4H, td, CH_{Ar}), 8.03; 8.07 (4H, dd, CH_{Ar}); 7.9 (1H, s, –NH); 7.07–7.27 (5H, m, CH_{Ar}); 3.55–3.58 (2H, m, –OCH_2); 2.35–2.41 (1H, m, CH); 0.82–0.84 (6H, dt, $2CH_3$)	3175(–NH–), 1654 (C=O), 1520 (Ar), 1381 ($C(CH_3)_2$)
18d[a]	$C_{22}H_{15}NSO_3$ 147	7.66; 7.56 (4H, td, CH_{Ar}), 8.12; 8.05 (4H, dd, CH_{Ar}); 7.95 (1H, s broad, –NH); 7.01–7.44 (10H, m broad, CH_{Ar})	3160 (–NH–), 1648 (C=O), 1610 (Ar)
19a[a]	$C_{17}H_{12}NClSO_3$ 135.5	7.66; 7.78 (4H, td, CH_{Ar}), 8.08; 8.02 (4H, dd, CH_{Ar}); 7.86 (1H, s, –NH); 7.36 (2H, d, CH_{Ar}); 7.01 (2H, d, CH_{Ar}); 2.84 (3H, s, –OCH_3)	3175 (–NH–), 2812 (OCH_3), 1658 (C=O), 1450 (Ar)
19b[a]	$C_{18}H_{14}NClSO_3$ 149	7.66; 7.56 (4H, td, CH_{Ar}), 8.12; 8.05 (4H, dd, CH_{Ar}); 7.89 (1H, s, –NH); 7.35 (2H, d, CH_{Ar}); 7.02 (2H, d, CH_{Ar}); 3.98 (2H, q, –OCH_2); 1.1 (3H, t, CH_3)	3170 (–NH–), 1651 (C=O), 1450 (Ar), 1374 (CH_3)
19c[a]	$C_{19}H_{16}NClSO_3$ 136–137	7.79; 7.83 (4H, td, CH_{Ar}), 8.03; 8.12 (4H, dd, CH_{Ar}); 7.75 (1H, s, –NH); 7.41 (2H, d, CH_{Ar}); 7.09 (2H, d, CH_{Ar}); 3.58–3.62 (2H, m, –OCH_2); 2.25–2.35 (1H, m, CH); 0.82–0.84 (6H, m, $2CH_3$)	3175 (–NH–), 1660 (C=O), 1510 (Ar), 1370 ($C(CH_3)_2$)
19d[a]	$C_{22}H_{14}NClSO_3$ 141	7.45; 7.51 (4H, td, CH_{Ar}), 7.97; 8.05 (4H, dd, CH_{Ar}); 7.94 (1H, s, –NH); 6.94–7.09 (4H, m, CH_{Ar}); 7.34–7.44 (5H, m, CH_{Ar})	3160 (–NH–), 1648 (C=O), 1600 (Ar)
20a[a]	$C_{11}H_7SClO_3$ 82	7.56; 7.68 (4H, td, CH_{Ar}), 8.02; 8.08 (4H, dd, CH_{Ar}); 2.82 (3H, s, –OCH_3)	2810 (OCH_3), 1652 (C=O)
20b[a]	$C_{12}H_9SClO_3$ 86	7.79; 7.65 (4H, td, CH_{Ar}), 8.21; 8.11 (4H, dd, CH_{Ar}); 3.98 (2H, t, CH_2); 1.20 (3H, t, CH_3)	1645 (C=O), 1380 (CH_3)
20c[a]	$C_{13}H_{11}SClO_3$ 79	7.68; 7.58 (4H, td, CH_{Ar}), 8.14; 8.09 (4H, dd, CH_{Ar}); 3.61–3.65 (2H, m, –OCH_2); 2.24–2.42 (1H, m, CH); 0.8–0.85 (6H, m, $2CH_3$)	1660 (C=O), 1370 ($C(CH_3)_2$)
20d[a]	$C_{16}H_9SClO_3$ 91	7.79; 7.64 (4H, td, CH_{Ar}), 8.11; 8.05 (4H, dd, CH_{Ar}); 7.06–7.28 (5H, m, CH_{Ar})	1648 (C=O), 1450 (Ar)
21a[a]	$C_{12}H_{10}S_2O_4$ 131	7.64; 7.54 (4H, td, CH_{Ar}), 8.09; 8.05 (4H, dd, CH_{Ar}); 2.79 (3H, s broad, –OCH_3)	2815 (OCH_3), 1650 (C=O)
21b[a]	$C_{14}H_{14}S_2O_4$ 127	7.49; 7.56 (4H, td, CH_{Ar}), 8.19; 8.07 (4H, dd, CH_{Ar}); 3.93 (4H, q, –OCH_2); 1.14 (3H, t, CH_3)	1648 (C=O), 1440 (CH_3)
21c[a]	$C_{16}H_{18}S_2O_4$ 135	7.68; 7.59 (4H, td, CH_{Ar}), 8.14; 8.07 (4H, dd, CH_{Ar}); 3.57–3.60 (4H, m, $2OCH_2$); 2.28–2.43 (1H, m, CH); 0.85–0.91 (6H, m, $2CH_3$)	1660 (C=O), 1365 ($C(CH_3)_2$)
21d[a]	$C_{22}H_{14}S_2O_4$ 145	7.77; 7.62 (4H, td, CH_{Ar}), 8.09; 8.03 (4H, dd, CH_{Ar}); 7.03–7.34 (10H, m broad, CH_{Ar})	1648 (C=O), 1510 (Ar)
22a[b]	$C_{15}H_{14}NClSO_2$ 88	7.45; 7.51 (4H, td, CH_{Ar}), 8.14; 8.09 (4H, dd, CH_{Ar}); 1.97–2.41 (4H, m, –$N(CH_2)_2$); 1.08–1.57 (6H, m, $3CH_2$)	1661 (C=O), 1350 (*tert*-N)
22b[b]	$C_{14}H_{12}NClSO_3$ 101	7.79; 7.83 (4H, td, CH_{Ar}), 8.11; 8.02 (4H, dd, CH_{Ar}); 3.54–3.7 (4H, m, –$N(CH_2)_2$); 2.34–2.49 (4H, m, –$O(CH_2)_2$)	1658 (C=O), 1355 (*tert*-N)
22c[b]	$C_{16}H_{16}NClSO_2$ 118	7.66; 7.58 (4H, td, CH_{Ar}), 8.12; 8.06 (4H, dd, CH_{Ar}); 7.24–7.54 (5H, m, CH_{Ar}); 7.82 (1H, s, –NH)	3160 (–NH–), 1648 (C=O), 1500 (Ar)
23a[b]	$C_{15}H_{16}N_2SO_2$ 93	7.68; 7.59 (4H, td, CH_{Ar}), 7.97; 8.05 (4H, dd, CH_{Ar}); 6.72 (2H, s, NH_2); 1.96–2.37 (4H, m, –$N(CH_2)_2$); 1.08–1.54 (6H, m, $3CH_2$)	1651 (C=O), 1358 (*tert*-N)
23b[b]	$C_{14}H_{14}N_2SO_3$ 124–125	7.77; 7.62 (4H, td, CH_{Ar}), 8.11; 8.08 (4H, dd, CH_{Ar}); 6.8 (2H, s, NH_2); 3.56–3.72 (4H, m, –$N(CH_2)_2$); 3.56–3.72 (4H, m, –$O(CH_2)_2$)	1661 (C=O), 1340 (*tert*-N)
23c[b]	$C_{16}H_{12}N_2SO_2$ 145	7.79; 7.65 (4H, td, CH_{Ar}), 7.99; 8.02 (4H, dd, CH_{Ar}); 6.83 (2H, s, NH_2); 7.24–7.32 (5H, m, CH_{Ar})	3160 (–NH–), 1660 (C=O), 1350 (*tert*-N), 1510 (Ar)

(Continued)

TABLE 4 Continued

	Formula, mp (°C)	*^1H (δ, ppm)*	*IR (cm^{-1})*
24a[b]	$C_{19}H_{24}N_2SO_2$ 78	7.66; 7.78 (4H, td, CH_{Ar}), 8.21; 8.11 (4H, dd, CH_{Ar}); 3.44–3.49 (4H, m, $-N(CH_2)_2$); 1.96–2.41 (4H, m, $-N(CH_2)_2$); 1.44–1.57 (4H, m, $-O(CH_2)_2$); 1.12 (6H, s, $2CH_3$)	1654 (C=O), 1340(*tert*-N)
24b[b]	$C_{18}H_{22}N_2SO_3$ 99–101	7.68; 7.58 (4H, td, CH_{Ar}), 8.02; 8.07 (4H, dd, CH_{Ar}); 3.57–3.68 (4H, m, $-O(CH_2)_2$); 3.48–3.52 (4H, m, $-N(CH_2)_2$); 2.36–2,47 (4H, m, $-N(CH_2)_2$); 1.21 (6H, s, $2CH_3$)	1645 (C=O), 1355 (*tert*-N)
24c[b]	$C_{20}H_{20}N_2SO_2$ 116	7.66; 7.58 (4H, td, CH_{Ar}), 8.06; 8.12 (4H, dd, CH_{Ar}); 7.24–7.54 (5H, m, CH_{Ar}); 3.48 (4H, q, $-N(CH_2)_2$); 1.18 (6H, s, $2CH_3$)	3170 (–NH–), 1656 (C=O), 1590 (Ar)
25a[b]	$C_{23}H_{32}N_2SO_2$ 91	7.47; 7.56 (4H, td, CH_{Ar}), 7.98; 8.02 (4H, dd, CH_{Ar}); 3.09–3.12 (4H, q, $-N(CH_2)_2$); 1.96–2.41 (4H, m, $-N(CH_2)_2$); 1.12–1.35 (8H, q, $4CH_2$); 0.86–0.91 (6H, m, $2CH_3$)	1651 (C=O), 1375 (CH_3), 1355 (*tert*-N)
25b[b]	$C_{22}H_{30}N_2SO_3$ 121	7.73; 7.64 (4H, td, CH_{Ar}), 8.02; 8.05 (4H, dd, CH_{Ar}); 3.09–3.11 (4H, m, $-N(CH_2)_2$); 2.35–2.47 (4H, m, $-N(CH_2)_2$); 1.24–1.34 (8H, m, $4CH_2$); 0.84–0.94 (6H, m, $2CH_3$); 3.55–3.59 (4H, m, $-O(CH_2)_2$)S	1659 (C=O), 1381 (CH_3), 1340 (*tert*-N)
25c[b]	$C_{24}H_{28}N_2SO_2$ 136	7.66; 7.56 (4H, td, CH_{Ar}), 8.22; 8.10 (4H, dd, CH_{Ar}); 7.24–7.54 (5H, m, CH_{Ar}); 3.09–3.11 (4H, q, $-N(CH_2)_2$); 1.24–1.38 (8H, q, $4CH_2$); 0.84–0.94 (6H, m, $2CH_3$)	3163 (–NH–), 1660 (C=O), 1580 (Ar)
26a[b]	$C_{20}H_{24}N_2SO_2$ 112	7.56; 7.62 (4H, td, CH_{Ar}), 8.03; 8.07 (4H, dd, CH_{Ar}); 1.95–3.2 (8H, m, $-N(CH_2)_2$); 1.08–1.56 (12H, q, $6CH_2$)	1645 (C=O), 1360 (*tert*-N)
26b[b]	$C_{19}H_{22}N_2SO_3$ 124	7.47; 7.65 (4H, td, CH_{Ar}), 8.02; 8.06 (4H, dd, CH_{Ar}); 3.7–3.83 (4H, m, $-N(CH_2)_2$); 3.55–3.68 (4H, m, $-O(CH_2)_2$); 2.35–2.47 (4H, m, $-N(CH_2)_2$); 1.29–1.47 (6H, m, $2CH_3$)	1655 (C=O), 1351 (*tert*-N)
26c[b]	$C_{21}H_{20}N_2SO_2$ 126	7.98; 8.02 (4H, td, CH_{Ar}), 8.06; 8.12 (4H, dd, CH_{Ar}); 7.24–7.53 (5H, m, CH_{Ar}); 3.73–3.82 (4H, m, $-N(CH_2)_2$); 1.32–1.4 (6H, m, $2CH_3$)	3160 (–NH–), 1662 (C=O), 1500 (Ar)
27a[b]	$C_{19}H_{22}N_2SO_3$ 89	7.79; 7.65 (4H, td, CH_{Ar}), 8.02; 8.05 (4H, dd, CH_{Ar}); 3.55–3.67 (4H, m, $-N(CH_2)_2$); 3.38–3.45 (4H, m, $-O(CH_2)_2$); 1.96–2.3 (4H, m, $-N(CH_2)_2$); 1.1–1.25 (6H, m, $3CH_2$)	1652 (C=O), 1351 (*tert*-N)
27b[b]	$C_{18}H_{20}N_2SO_4$ 114–115	7.73; 7.64 (4H, td, CH_{Ar}), 8.02; 8.07 (4H, dd, CH_{Ar}); 3.53 (16H, m, $CH_{2morpholine}$)	1660 (C=O), 1340 (*tert*-N)
27c[b]	$C_{20}H_{18}N_2SO_3$ 129	7.73; 7.64 (4H, td, CH_{Ar}), 8.11; 8.02 (4H, dd, CH_{Ar}); 7.26–7.42 (5H, m, CH_{Ar}); 3.49 (8H, m, $CH_{2morpholine}$)	1648 (C=O), 1500 (Ar), 1351 (*tert*-N)
28a[b]	$C_{21}H_{20}N_2SO_2$ 109	7.45; 7.51 (4H, td, CH_{Ar}), 8.14; 8.09 (4H, dd, CH_{Ar}); 7.07–7.3 (5H, m, CH_{Ar}); 6.3 (1H, s, –NH); 1.96–2.41 (4H, m, $-N(CH_2)_2$); 1.09–1.52 (6H, m, $3CH_2$)	3161(–NH–), 1650 (C=O), 1600 (Ar), 1350 (*tert*-N)
28b[b]	$C_{20}H_{18}N_2SO_3$ 123	7.49; 7.56 (4H, td, CH_{Ar}), 8.19; 8.07 (4H, dd, CH_{Ar}); 7.08–7.29 (5H, m, CH_{Ar}); 6.78 (1H, s, –NH); 3.55–3.7 (4H, m, $-O(CH_2)_2$); 2.34–2.46 (4H, m, $-N(CH_2)_2$)	3175 (–NH–), 1645 (C=O), 1580 (Ar), 1340 (*tert*-N)
28c[b]	$C_{22}H_{16}N_2SO_2$ 141	7.66; 7.56 (4H, td, CH_{Ar}), 8.12; 8.05 (4H, dd, CH_{Ar}); 7.07–7.54 (10H, m, CH_{Ar}); 6.85 (1H, s, –NH)	3171 (–NH–), 1662 (C=O), 1510 (Ar),
29a[b]	$C_{21}H_{19}N_2SO_2Cl$ 116	7.68; 7.61 (4H, td, CH_{Ar}), 8.22; 8.15 (4H, dd, CH_{Ar}); 6.89 (1H, s, –NH); 7.08 (2H, d, CH_{Ar}); 7.36 (2H, d, CH_{Ar}); 1.96–2.41 (4H, m, $-N(CH_2)_2$); 1.09–1.52 (6H, m, $3CH_2$)	3160 (–NH–), 1650 (C=O), 1580 (Ar), 1350 (*tert*-N)
29b[b]	$C_{20}H_{17}N_2SO_3Cl$ 129	7.73; 7.64 (4H, td, CH_{Ar}), 8.06; 8.12 (4H, dd, CH_{Ar}); 7.75 (1H, s, –NH); 7.08 (2H, d, CH_{Ar}); 7.35 (2H, d, CH_{Ar}); 3.58–3.67 (4H, m, $-O(CH_2)_2$); 2.34–2.49 (4H, m, $-N(CH_2)_2$)	3162 (–NH–), 1660 (C=O), 1575 (Ar), 1360 (*tert*-N)
29c[b]	$C_{22}H_{15}N_2SO_2Cl$ 151	7.98; 8.02 (4H, td, CH_{Ar}), 8.12; 8.05 (4H, dd, CH_{Ar}); 7.13–7.54 (9H, m broad, CH_{Ar}); 6.98 (1H, s, –NH)	3175 (–NH–), 1648 (C=O), 1575 (Ar)
30a[b]	$C_{20}H_{24}N_2S_2O_2$ 143	7.79; 7.65 (4H, td, CH_{Ar}), 7.99; 8.01 (4H, dd, CH_{Ar}); 1.96–2.41 (8H, m broad, $-N(CH_2)_2$); 1.09–1.52 (12H, m broad, $6CH_2$)	1650 (C=O), 1358 (*tert*-N)
30b[b]	$C_{18}H_{20}N_2S_2O_4$ 164	7.41; 7.53 (4H, td, CH_{Ar}), 8.17; 8.11 (4H, dd, CH_{Ar}); 2.34–2.48 (8H, m broad, $-N(CH_2)_2$); 3.55–3.68 (8H, m broad, $-O(CH_2)_2$)	1658 (C=O), 1360 (*tert*-N)
30c[b]	$C_{22}H_{16}N_2S_2O_2$ 158	7.63; 7.51 (4H, td, CH_{Ar}), 8.10; 8.02 (4H, dd, CH_{Ar}); 7.23–7.54 (8H, m broad, CH_{Ar}); 6.89 (2H, s broad, –NH)	3175 (–NH–), 1661 (C=O), 1575 (Ar)

^{1}H NMR solvents: [a]$CHCl_3$; [b]DMSO.

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