

## Unexpected reaction of *N,N*-dichloroarenesulfonamides with divinyl sulfide: formation of *N*-[2-chloro- and *N*-[2,2-dichloro-1-(arylsulfonylamino)ethyl]arenesulfonamides

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### Abstract

Reaction of *N,N*-dichloroarenesulfonamides with divinyl sulfide was accompanied by cleavage of divinyl sulfide moiety and resulted in unexpected *N*-[2-chloro- and *N*-[2,2-dichloro-1-(arylsulfonylamino)ethyl]arenesulfonamides as final products. Possible reaction mechanism was discussed. The structure of bis(sulfonamido)chloroethanes synthesized was studied by X-ray analysis.

**Keywords:** *N,N*-dichlorosulfonamides, divinyl sulfide, sulfonamides, X-ray analysis

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### Introduction

*N,N*-Dichlorosulfonamides are important and available reagents in modern organic and elemento-organic synthesis.<sup>1-22</sup> A high reactivity of *N,N*-dichlorosulfonamides is caused by the ability of N–Cl group to enter into a homolytic or heterolytic cleavage depending on conditions to produce sulfonylamidyl radicals and atomic chlorine, sulfonylamidyl anions and cations of chlorine or nitrenes. *N,N*-Dichlorosulfonamides, in contrast to *N,N*-dichloroacylamides or *N,N*-dichlorourethanes are more stable under keeping but at the same time are more selective and reactive<sup>14,23</sup> in reactions because of strong electro-withdrawing nature of sulfonyl group and weak conjugation between SO<sub>2</sub> group and nitrogen atom. Due to such features *N,N*-dichlorosulfonamides are useful for diverse chemical transformations. Reaction of *N,N*-dichlorosulfonamides with alkenes is an efficient method for the synthesis of *N*-( $\beta$ -

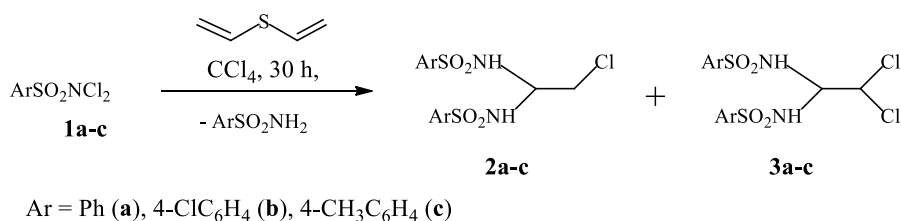
chloroalkyl)sulfonamides<sup>9-13</sup> and *N*-sulfonylpolyhaloaldimines<sup>14-19</sup> which were used as key reagents in further preparation of a large range of heterocyclic and open chained functionally substituted sulfonamide derivatives.<sup>10,14-19,24-27</sup> In elemento-organic chemistry oxidative imination of heteroatomic compounds, and mainly sulfur containing reagents, are of great significance.<sup>1,2,20-22</sup>

Divinyl sulfide is an important representative of practically useful sulfur organic compounds. The efficient method for the synthesis of divinyl sulfide was developed on the basis of acetylene and sodium sulfide,<sup>28-31</sup> which are available starting materials. Divinyl sulfide is a multipurpose reagent in modern organic chemistry, for it is applied in synthesis of different acyclic and heterocyclic sulfur containing derivatives.<sup>28-39</sup> Divinyl sulfide is a high-performance synthon of vinylthio group<sup>39</sup> and it is active in reactions with both radical and electrophilic reagents.<sup>28-39</sup> The utilization of divinyl sulfide as cross-link monomer was used for the synthesis of new polymer products and ion exchange resins possessing unique properties.<sup>29,30</sup> A divinyl sulfide cyclization with thio- or selenourea in the presence of strong acids results in the formation of new heterocycles – dithiazine or thiaselenoazine derivatives.<sup>29-32</sup>

Though divinyl sulfide was widely used in different reactions,<sup>25-39</sup> it was never studied in reaction with *N,N*-dichloroamides. Only oxidative imination and chlorination of 2,2-dichlorovinyl sulfides by *N,N*-dichlorobenzensulfonamide are known.<sup>40</sup> And in the present paper we studied the reaction of divinyl sulfide with *N,N*-dichloroarenesulfonamide **1a-c** which are available typical representatives of *N,N*-dichlorosulfonamides.

## Results and Discussion

It was expected that the reaction of divinyl sulfide with dichloroamides **1a-c** can lead to products of chlorination, imination of sulfur atom or chloroamidation of double bonds, similar to the transformation of dichlorovinyl sulfide.<sup>40</sup> Unexpectedly the process was found to result in the formation of *N*-[2-chloro-1-(arylsulfonylamino)ethyl]arenesulfonamides **2a-c** and *N*-[2,2-dichloro-1-(arylsulfonylamino)ethyl]arenesulfonamides **3a-c** as the final compounds (Scheme 1). Yield of diamides **2a-c** and **3a-c** depended on both conditions and the order of the reagent mixing.



**Scheme 1.** Reaction of dichloroamides **1a-c** with divinyl sulfide.

The influence of the reaction conditions was studied in detail with divinyl sulfide and dichloroamide **1b** (Table 1). The reaction was carried out in CCl<sub>4</sub> which does not react with dichloroamide **1b**. The process was exothermic. A strong resinification was observed when the reagents were mixed without cooling and 4-chlorobenzensulfonamide was isolated as the only identified product in this case.

**Table 1.** Yield of bis-amides **2b** and **3b** in the reaction of divinyl sulfide with dichloroamide **1b** under various conditions<sup>a</sup>

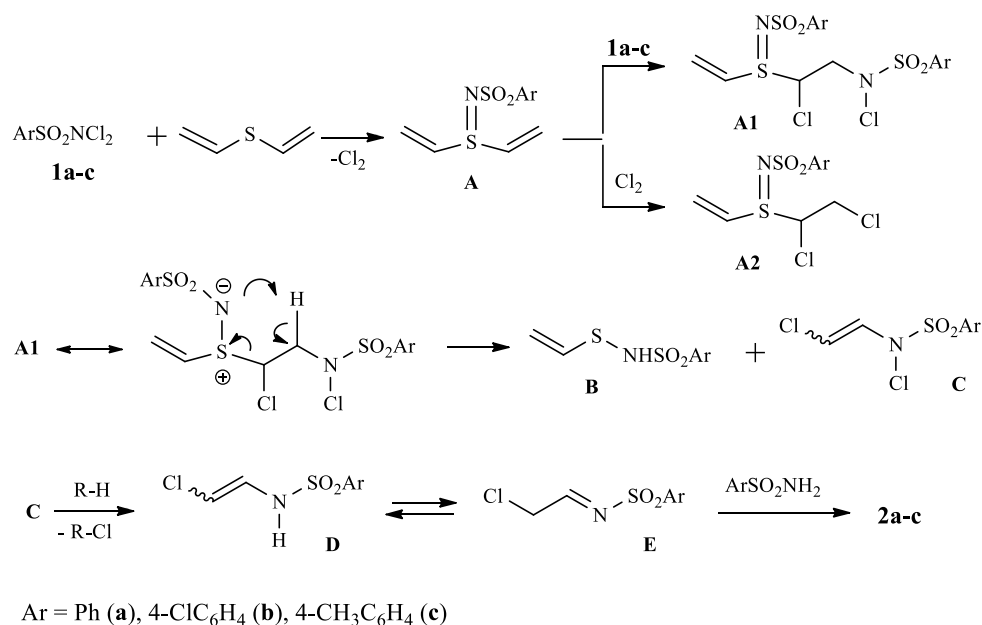
Entry	Ratio of divinyl sulfide : <b>1b</b>	Temperature, °C	Volume of CCl <sub>4</sub> , mL	Yield, %	
				<b>2b</b>	<b>3b</b>
1 <sup>b</sup>	0.50 g (5.8 mmol): 3.02 g (11.6 mmol)	50	40	0	0
2 <sup>b</sup>	0.50 g (5.8 mmol): 3.02 g (11.6 mmol)	0	40	23	21
3 <sup>b</sup>	0.50 g (5.8 mmol) : 6.04 g (23.2 mmol)	0	40	35	5
4 <sup>b</sup>	0.50 g (5.8 mmol) : 3.02 g (11.6 mmol)	-10	40	29	25
5 <sup>b</sup>	0.50 g (5.8 mmol) : 6.04 g (23.2 mmol)	-10	40	48	9
6 <sup>b</sup>	0.50 g (5.8 mmol) : 3.02 g (11.6 mmol)	-40	40	37	30
7 <sup>c</sup>	0.50 g (5.8 mmol) : 3.02 g (11.6 mmol)	-40	100	65	28
8 <sup>c</sup>	0.50 g (5.8 mmol) : 6.04 g (23.2 mmol)	-40	100	69	0
9 <sup>d</sup>	0.50 g (5.8 mmol) : 3.02 g (11.6 mmol)	-40	100	0	0
10 <sup>d</sup>	0.50 g (5.8 mmol) : 6.04 g (23.2 mmol)	-40	100	0	0

<sup>a</sup>The reaction mass was stirred at the appropriate temperature for 30 h to give a precipitate of the products. Resinification of the reaction mass and formation of 4-chlorobenzensulfonamide took place in any cases. <sup>b</sup>Solution of dichloroamide **1b** in 20 mL CCl<sub>4</sub> was added to solution of divinyl sulfide in 20 mL CCl<sub>4</sub>. <sup>c</sup>Solution of dichloroamide **1b** in 50 mL CCl<sub>4</sub> was added to solution of divinyl sulfide in 50 mL CCl<sub>4</sub>. <sup>d</sup>Solution of divinyl sulfide in 20 mL CCl<sub>4</sub> was added to solution of dichloroamide **1b** in 80 mL CCl<sub>4</sub>.

The yield of bis-amides **2b**, **3b** was found to depend on both ratio and concentration of the reagents (Table 1). A dilution of the reaction mixture assisted to increasing of the yield of **2b** to 69% when 4 equivalents of dichloroamide **1b** were used (Table 1, Entry 8) and no compound **3b** was produced under such conditions. At a higher concentration of the reagents bis-amide **3b** was produced in the highest yield (30%) when 2 equivalents of dichloroamide **1b** were added to cooled solution of divinyl sulfide (Table 1, Entry 6). When divinyl sulfide was added to a solution of dichloroamide **1b** (Table 1, Entries 9, 10) diamides **2b**, **3b** were not obtained and complicated mixture of unidentified resin-like products of variable composition containing 21.45 – 23.16% of sulfur and 28.93 – 29.11% of chlorine was produced.

The bis-amides **2a-c** are produced apparently *via* intermediates **A** and **A1** which are formed after oxidative imination and chloroamidation of divinyl sulfide with dichloroamides **1a-c** (Scheme 2). It may be suggested that intermediates **A1** are subjected to further cleavage to

produce vinylsulfenamides **B** and *N*-chloroenamides **C**. Such elimination of an unsaturated molecule is probably similar to reaction of sulfonium ylides containing a vinyl or ethynyl group.<sup>41</sup> *N*-Chloroenamides **C** are able to be reduced into enamides **D** which turn into tautomers **E** followed by addition of 4-arenesulfonamide to C=N group to give the final bis-amides **2a-c** (Scheme 2).



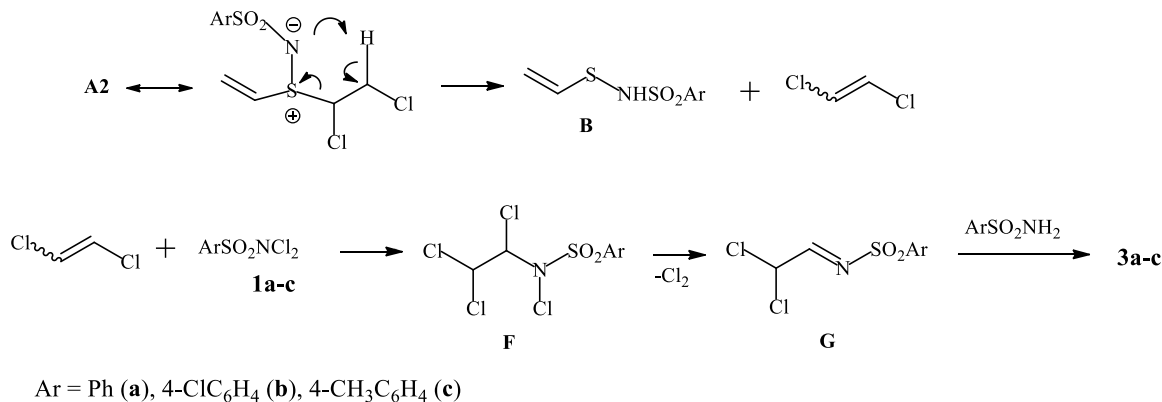
**Scheme 2.** A possible mechanism for the formation of bis-amides **2a-c**.

It is not inconceivable that bis-amides **3** are produced from intermediates **A2** after imination and chlorination of divinyl sulfide (Schemes 2, 3). Taking into account the possibility of an olefin elimination from a vinyl containing sulfonium ylide system<sup>41</sup> it may be suggested that intermediates **A2** are able to eliminate 1,2-dichloroethylene. 1,1-Bis(amido)-2,2-dichloroethanes are known to be formed in reaction of *N,N*-dichloroamides **1** with 1,2-dichloroethylene.<sup>14, 42</sup> It was assumed that the reaction proceeded *via* saturated *N*-chloro- adducts **F** followed by easy dechlorination resulting in *N*-sulfonyldichloroacetaldimines **G**.<sup>13</sup> Then arenesulfonamides are added to intermediate imines **G** (Scheme 3). Nucleophilic addition of arenesulfonamides to haloaldehydes has been previously reviewed in detail.<sup>14</sup>

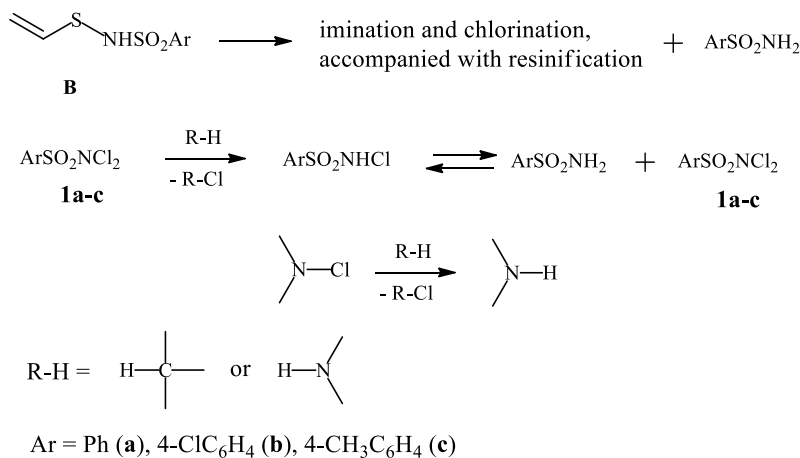
Probable vinylsulfenamides **B** produced both from intermediates **A1** and **A2** (Schemes 2, 3) obviously undergo further imination and chlorination accompanied by resinification of the reaction mass and formation of arenesulfonamides.

Reduction of the probable *N*-Cl intermediates and starting dichloroamides **1a-c** into *N*-H derivatives is possible in reaction with different compounds containing C-H or N-H fragments which are present in the reaction mixture (Scheme 4).

Judging from the reaction course supported (Schemes 2-4), it is evident that increasing the ratio and concentration of dichloroamides **1a-c** gives rise to increase probability of intermediates **A1** formation (Scheme 2). Therefore yield of diamides **2a-c**, produced from intermediates **A1**, is expected to be higher in this case. Under small ratio of dichloroamides **1a-c** to divinyl sulfide probability of intermediates **A1** formation decreases, yield of diamides **2a-c** expected to be less, and in this case the second reaction course resulting in diamides **3a-c** via intermediates **A2** (Scheme 3) can be more significant.



**Scheme 3.** A possible mechanism for the formation of bis-amides **3a-c**.

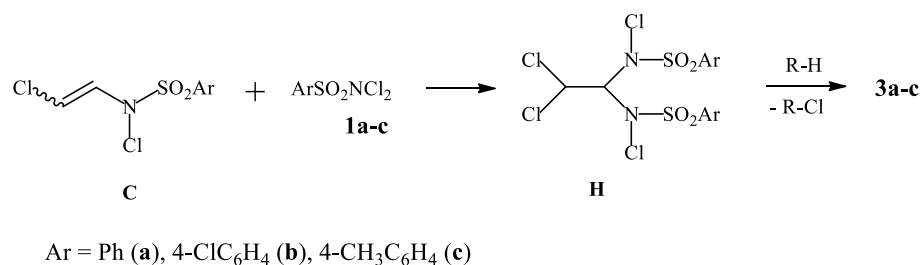


**Scheme 4.** Proposed formation of N-H species in the reaction.

This conclusion is in accordance with experimental data shown in Table 1. In real situation yield of diamide **2b** was much higher than yield of **3b** when we used 4 equivalents of dichloroamide **1b** (Table 1, Entries 3, 5, 8). The increased content of diamide **3b** in the mixture of the products was found to be for the less quantity of dichloroamide **1b** (Table 1, Entries 2, 4, 6, 7).

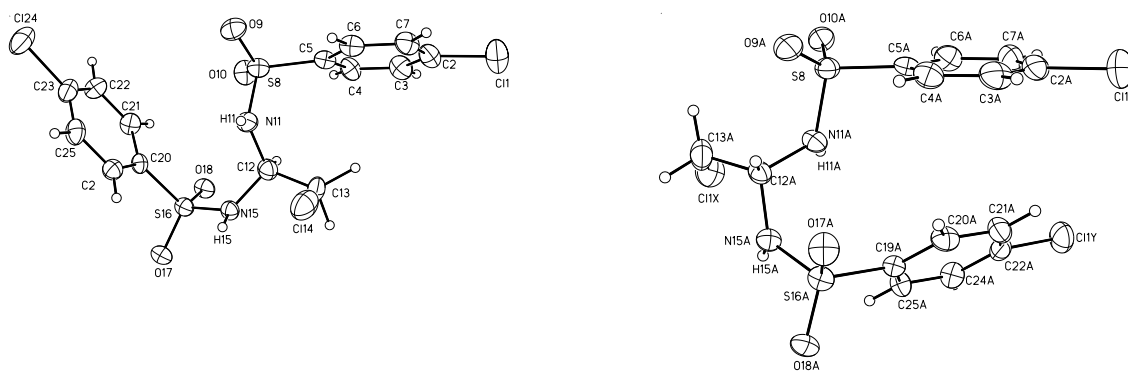
Dilution of the reaction mixture led to increase in the yield of diamides **2b**, **3b** (Table 1, Entries 7, 8) probably because of less activity of side resinification process. Very large concentration and excess of dichloroamide **1b** resulted in comprehensive resinification, and diamides **2b**, **3b** were not isolated at all as this took place when divinyl sulfide was added into excess of dichloroamide **1b** (Entries 9, 10).

Other possible ways for the formation of the products are not ruled out. For instance, diamides **3a-c** can be produced from the dichloroamides **1a-c** and intermediates **C** (formed according to Scheme 3) *via* adducts **H** followed by reduction of N-Cl groups (Scheme 5).

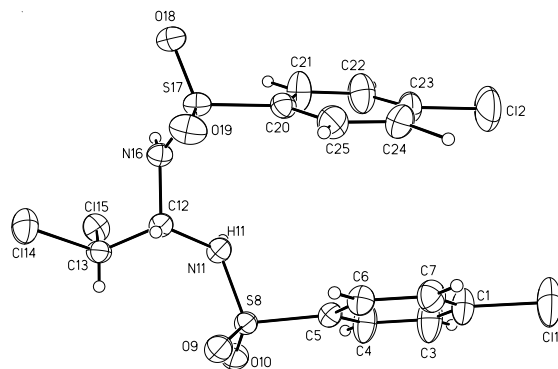


**Scheme 5.** The possible formation of bis-amides **3a-c** via enamides **C** and adducts **H**.

Unequivocal determination of the structures of diamides **2b**, **3b** was made by X-ray diffraction. The necessary single crystal samples were obtained by slowly evaporating solutions of **2b** and **3b** in the mixture of CH<sub>2</sub>Cl<sub>2</sub> with *n*-heptane. It was found that the crystal structure of **2b** is formed by two crystallographically independent molecules having different conformations. Molecules of every type are connected together through H-bonds (N-H...O(S), N...O 2.831(4)–2.975(4) Å) into the infinite bands. All other contacts are comparable in length to the sum of the van der Waals radii of corresponding atoms. H-bonds of the same type link molecules of **3b** into framework (Figures 1, 2).



**Figure 1.** Two crystallographically independent molecules of bis-amide **2b**.



**Figure 2.** Structure of the bis-amide **3b** molecule.

## Conclusions

Thus, the first example of unusual reaction of *N,N*-dichlorosulfonamides with divinyl sulfide was demonstrated. Unexpected 1,1-bis(sulfonamido)-2-chloroethanes **2a-c** and 1,1-bis(sulfonamido)-2,2-dichloroethanes **3a-c** were obtained. The influence of conditions and ratio of the reagents on the reaction course and yield of diamides **2a-c**, **3a-c** was shown. The structure of diamides **2a-c**, **3a-c** was studied by spectral methods and X-ray analysis. The possible reaction way for the formation of compounds **2a-c** and **3a-c** was discussed.

1,1-Bis(sulfonamido)-2-chloroethanes **3** are synthetically attractive as *C*-amidoalkylating reagents in reactions with aromatics and heteroaromatics.<sup>43</sup> In addition, owing to NH and CH<sub>2</sub>Cl or CHCl<sub>2</sub> groups in their structures, compounds **2a-c**, **3a-c** may be thought of as evident precursors of heterocyclic derivatives and ligands.

## Experimental Section

**General.** *N,N*-Dichloro-arenesulfonamides **1a-c** were obtained by chlorination of arenesulfonamides<sup>15</sup> and purified by recrystallization from CCl<sub>4</sub>. Divinyl sulfide was synthesized by the method.<sup>28</sup> NMR spectra were recorded on a Bruker DPX 400 spectrometer (<sup>1</sup>H, 400.13 MHz; <sup>13</sup>C, 100.61 MHz) at 25 °C with HMDS as an internal standard. Chemical shifts are reported in ppm (δ), and coupling constants (*J*) in Hz. IR spectra were recorded on a Bruker IFS-25 spectrophotometer in KBr. All melting points were measured on a Kofler micro hot stage apparatus. Elemental analyses for C, H and N were obtained using a Thermo Finnigan Flash series1112 EA analyzer. Single crystal diffraction data for the compounds were collected on a SMART APEX II CCD (Bruker AXS) automatic diffractometer (Mo-*K*α, λ = 0.71073 Å, T = 240 K); an absorption correction was applied using the Bruker SADABS program, version 2.10. The structures were solved by direct methods and refined by the full-matrix least-squares method

in an anisotropic approximation for non-hydrogen atoms. The H atoms were calculated geometrically and used in riding model refinement. All structure solution and refinement calculations were carried out with SHELX-97 and Bruker SHELXTL Version 6.14 program packages. Crystallographic data (excluding structure factors) for the structures in this letter have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication numbers CCDC (**2** – 816999, **3** – 817000). Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK [fax: +44(0)-1223-336033 or e-mail: deposit@ccdc.cam.ac.uk].

**General experimental procedure for the synthesis of compounds 2, exemplified by *N*-{2-chloro-1-[(phenylsulfonyl)amino]ethyl}benzenesulfonamide (2a)**

A solution of *N,N*-dichlorobenzenesulfonamide **1a** (5.24 g, 23.2 mmol) in 50 mL CCl<sub>4</sub> was added dropwise over 25 min. to a magnetically stirred solution of divinyl sulfide (0.50 g, 5.8 mmol) in 50 mL CCl<sub>4</sub> at –40 °C. The reaction mass was stirred for 30 h at –40 °C, a precipitate obtained was separated, rinsed out with 50 ml of diethyl ether, and recrystallized from hexane to give compound **2a**.

**2a.** Colorless crystals, yield 67%, 1.46 g, mp 134–135 °C, IR ( $\nu_{\max}$ , KBr, cm<sup>-1</sup>): 1165, 1340 (SO<sub>2</sub>), 3260 (NH). <sup>1</sup>H NMR (400.13 MHz, DMSO-*d*<sub>6</sub>),  $\delta_{\text{H}}$  3.45 (2H, d, <sup>3</sup>*J*<sub>HH</sub> = 6.5 Hz, CH<sub>2</sub>Cl), 4.87 (1H, tt, <sup>3</sup>*J*<sub>HH</sub> = 6.5 Hz, <sup>3</sup>*J*<sub>HH</sub> = 8.3 Hz, NCH), 7.31, 7.78 (10H, m, C<sub>6</sub>H<sub>5</sub>), 7.80 (2H, d, <sup>3</sup>*J*<sub>HH</sub> = 8.3 Hz, NH). <sup>13</sup>C NMR (100.61 MHz, DMSO-*d*<sub>6</sub>),  $\delta_{\text{C}}$  47.5 (CH<sub>2</sub>Cl), 63.8 (CH), 125.4, 126.3, 132.7, 133.1 (C<sub>6</sub>H<sub>5</sub>). Anal. Calcd for C<sub>14</sub>H<sub>15</sub>ClN<sub>2</sub>O<sub>4</sub>S<sub>2</sub> (374.86): C, 44.86; H, 4.03; Cl, 9.46; N, 7.47; S, 17.11%. Found: C, 44.38; H, 3.99; Cl, 9.24; N, 7.38; S, 17.45%.

**4-Chloro-*N*-(2-chloro-1-[(4-chlorophenyl)sulfonyl]amino)ethyl)benzenesulfonamide (2b).**

Colorless crystals, yield 69%, 1.78 g, mp 148–149 °C, IR ( $\nu_{\max}$ , KBr, cm<sup>-1</sup>): 1160, 1337 (SO<sub>2</sub>), 3256 (NH). <sup>1</sup>H NMR (400.13 MHz, DMSO-*d*<sub>6</sub>),  $\delta_{\text{H}}$  3.68 (2H, d, <sup>3</sup>*J*<sub>HH</sub> 6.6 Hz, CH<sub>2</sub>Cl), 5.03 (1H, tt, <sup>3</sup>*J*<sub>HH</sub> 6.6 Hz, <sup>3</sup>*J*<sub>HH</sub> 8.2 Hz, CH), 7.90, 8.04 (8H, AA'BB', <sup>3</sup>*J*<sub>AB</sub> = <sup>3</sup>*J*<sub>A'B'</sub> = 8.7 Hz, <sup>4</sup>*J*<sub>AA'</sub> = 4.6 Hz, <sup>4</sup>*J*<sub>BB'</sub> = 4.2 Hz, 4-ClC<sub>6</sub>H<sub>4</sub>), 8.80 (2H, d, <sup>3</sup>*J*<sub>HH</sub> 8.2 Hz, NH). <sup>13</sup>C NMR (100.61 MHz, DMSO-*d*<sub>6</sub>),  $\delta_{\text{C}}$  44.1 (CH<sub>2</sub>Cl), 63.3 (CH), 126.1, 126.7, 127.3, 138.7 (C<sub>6</sub>H<sub>4</sub>). Anal. Calcd for C<sub>14</sub>H<sub>13</sub>Cl<sub>3</sub>N<sub>2</sub>O<sub>4</sub>S<sub>2</sub> (443.75): C, 37.89; H, 2.95; Cl, 23.97; N, 6.31; S, 14.45%. Found: C, 37.75; H, 3.02; Cl, 23.84; N, 6.28; S, 14.68%.

***N*-(2-Chloro-1-[(4-methylphenyl)sulfonyl]amino)ethyl)-4-methylbenzenesulfonamide (2c).**

Colorless crystals, yield 59%, 1.38 g, mp 169–170 °C, IR ( $\nu_{\max}$ , KBr, cm<sup>-1</sup>): 1170, 1345 (SO<sub>2</sub>), 3268 (NH). <sup>1</sup>H NMR (400.13 MHz, DMSO-*d*<sub>6</sub>),  $\delta_{\text{H}}$  2.34 (6H, s, CH<sub>3</sub>), 3.44 (2H, d, <sup>3</sup>*J*<sub>HH</sub> 6.6 Hz, CH<sub>2</sub>Cl), 4.82 (1H, tt, <sup>3</sup>*J*<sub>HH</sub> 6.6 Hz, <sup>3</sup>*J*<sub>HH</sub> 8.2 Hz, CH), 7.15, 7.66 (8H, AA'BB', <sup>3</sup>*J*<sub>AB</sub> = <sup>3</sup>*J*<sub>A'B'</sub> = 8.5 Hz, <sup>4</sup>*J*<sub>AA'</sub> = 4.5 Hz, <sup>4</sup>*J*<sub>BB'</sub> = 4.3 Hz, 4-MeC<sub>6</sub>H<sub>4</sub>), 8.46 (2H, d, <sup>3</sup>*J*<sub>HH</sub> 8.2 Hz, NH). <sup>13</sup>C NMR (100.61 MHz, DMSO-*d*<sub>6</sub>),  $\delta_{\text{C}}$  20.3 (CH<sub>3</sub>), 47.5 (CH<sub>2</sub>Cl), 63.7 (CH), 124.5, 125.9, 136.8, 139.0 (C<sub>6</sub>H<sub>4</sub>). Anal. Calcd for C<sub>16</sub>H<sub>19</sub>ClN<sub>2</sub>O<sub>4</sub>S<sub>2</sub> (402.92): C, 47.69; H, 4.75; Cl, 8.80; N, 6.95; S, 15.92%. Found: C, 47.92; H, 4.81; Cl, 8.62; N, 6.81; S, 15.56%.



**General experimental procedure for the synthesis of compounds 3, exemplified by *N*-{2,2-dichloro-1-[(phenylsulfonyl)amino]ethyl}benzenesulfonamide (3a)**

A solution of *N,N*-dichlorobenzene sulfonamide **1a** (2.62 g, 11.6 mmol) in 20 mL CCl<sub>4</sub> was added dropwise over 25 min. to a magnetically stirred solution of divinyl sulfide (0.50 g, 5.8 mmol) in 20 mL CCl<sub>4</sub> at -40 °C. The reaction mass was stirred for 30 h at -40 °C, a precipitate obtained was separated, rinsed out with 80 ml of diethyl ether, and recrystallized from CHCl<sub>3</sub> to give compound **3a**.

**3a.** Colorless crystals, yield 21%, 0.50 g, mp 143–144 °C, IR ( $\nu_{\max}$ , KBr, cm<sup>-1</sup>): 1175, 1340 (SO<sub>2</sub>), 3255 (NH). <sup>1</sup>H NMR (400.13 MHz, DMSO-*d*<sub>6</sub>),  $\delta_{\text{H}}$  5.15 (1H, dt, <sup>3</sup>*J*<sub>HH</sub> = 9.5 Hz, <sup>3</sup>*J*<sub>HH</sub> = 5.1 Hz, NCH), 5.92 (1H, d, <sup>3</sup>*J*<sub>HH</sub> = 5.1 Hz, CHCl<sub>2</sub>), 7.33, 7.65 (10H, m, C<sub>6</sub>H<sub>5</sub>), 9.11 (2H, d, <sup>3</sup>*J*<sub>HH</sub> = 9.5 Hz, NH). <sup>13</sup>C NMR (100.61 MHz, DMSO-*d*<sub>6</sub>),  $\delta_{\text{C}}$  68.7 (CHCl<sub>2</sub>), 74.0 (CH), 126.3, 128.9, 132.4, 141.4 (C<sub>6</sub>H<sub>5</sub>). Anal. Calcd for C<sub>14</sub>H<sub>14</sub>Cl<sub>2</sub>N<sub>2</sub>O<sub>4</sub>S<sub>2</sub> (409.31): C, 41.08; H, 3.45; Cl, 17.32; N, 6.84; S, 15.67%, Found: C, 41.27; H, 3.40; Cl, 17.58; N, 6.82; S, 15.88%.

**4-Chloro-*N*-(2,2-dichloro-1-[(4-chlorophenyl)sulfonyl]amino)ethyl}benzenesulfonamide**

**(3b).** Colorless crystals, yield 30%, 0.83 g, mp 169–170 °C, IR ( $\nu_{\max}$ , KBr, cm<sup>-1</sup>): 1165, 1334 (SO<sub>2</sub>), 3252 (NH). <sup>1</sup>H NMR (400.13 MHz, DMSO-*d*<sub>6</sub>),  $\delta_{\text{H}}$  5.17 (1H, dt, <sup>3</sup>*J*<sub>HH</sub> 4.3 Hz, <sup>3</sup>*J*<sub>HH</sub> 8.6 Hz, NCH), 6.07 (1H, d, <sup>3</sup>*J*<sub>HH</sub> 4.3 Hz, CHCl<sub>2</sub>), 7.51, 7.65 (8H, AA'BB', <sup>3</sup>*J*<sub>AB</sub> = <sup>3</sup>*J*<sub>A'B'</sub> = 8.6 Hz, <sup>4</sup>*J*<sub>AA'</sub> = 4.5 Hz, <sup>4</sup>*J*<sub>BB'</sub> = 4.2 Hz, 4-ClC<sub>6</sub>H<sub>4</sub>), 9.11 (2H, d, <sup>3</sup>*J*<sub>HH</sub> 8.6 Hz, NH). <sup>13</sup>C NMR (100.61 MHz, DMSO-*d*<sub>6</sub>),  $\delta_{\text{C}}$  68.8 (CHCl<sub>2</sub>), 74.2 (CH), 127.7, 128.3, 136.7, 143.1 (C<sub>6</sub>H<sub>4</sub>). Anal. Calcd for C<sub>14</sub>H<sub>12</sub>Cl<sub>4</sub>N<sub>2</sub>O<sub>4</sub>S<sub>2</sub> (478.20): C, 35.16; H, 2.53; Cl, 29.66; N, 5.86; S, 13.41%, Found: C, 35.07; H, 2.45; Cl, 29.48; N, 5.72; S, 13.69%.

***N*-(2,2-Dichloro-1-[(4-methylphenyl)sulfonyl]amino)ethyl-4-methylbenzenesulfonamide**

**(3c).** Colorless crystals, yield 37%, 0.94 g, mp 179–180 °C, IR ( $\nu_{\max}$ , KBr, cm<sup>-1</sup>): 1170, 1340 (SO<sub>2</sub>), 3260 (NH). <sup>1</sup>H NMR (400.13 MHz, DMSO-*d*<sub>6</sub>),  $\delta_{\text{H}}$  2.43 (6H, s, CH<sub>3</sub>), 5.23 (1H, dt, <sup>3</sup>*J*<sub>HH</sub> = 9.6 Hz, <sup>3</sup>*J*<sub>HH</sub> = 4.8 Hz, NCH), 5.96 (1H, d, <sup>3</sup>*J*<sub>HH</sub> = 4.8 Hz, CHCl<sub>2</sub>), 7.57, 7.76 (8H, AA'BB', <sup>3</sup>*J*<sub>AB</sub> = <sup>3</sup>*J*<sub>A'B'</sub> = 8.5 Hz, <sup>4</sup>*J*<sub>AA'</sub> = 4.5 Hz, <sup>4</sup>*J*<sub>BB'</sub> = 4.3 Hz, 4-MeC<sub>6</sub>H<sub>4</sub>), 8.83 (2H, d, <sup>3</sup>*J*<sub>HH</sub> = 9.6 Hz, NH). <sup>13</sup>C NMR (100.61 MHz, DMSO-*d*<sub>6</sub>),  $\delta_{\text{C}}$  21.0 (CH<sub>3</sub>), 68.7 (CHCl<sub>2</sub>), 74.1 (CH), 126.4, 129.2, 138.7, 142.6 (C<sub>6</sub>H<sub>4</sub>). Anal. Calcd for C<sub>16</sub>H<sub>18</sub>Cl<sub>2</sub>N<sub>2</sub>O<sub>4</sub>S<sub>2</sub> (437.36): C, 43.94; H, 4.15; Cl, 16.21; N, 6.41; S, 14.66%, Found: C, 43.85; H, 4.07; Cl, 16.30; N, 6.35; S, 14.78%.

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