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Sulfonamidation of Halogen-Substituted Electrophiles with *N*-(2,2,2-Trichloroethyl)arenesulfonamides

G. N. Chernysheva,* I. V. Nikitin, and I. B. Rozentsveig

Favorskii Irkutsk Institute of Chemistry, Siberian Branch, Russian Academy of Sciences, ul. Favorskogo 1, Irkutsk, 664033 Russia
**e-mail: gelya2010@irioch.irk.ru*

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Abstract—Reactions of *N*-(2,2,2-trichloroethyl)arenesulfonamides with primary alkyl bromides and iodides, allyl bromide, chloroacetonitrile, and benzyl chloride in acetonitrile on heating in the presence of potassium carbonate gave the corresponding *N*-alkyl-*N*-(2,2,2-trichloroethyl)arenesulfonamides.

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N-(Polychloroethyl) sulfonamides are promising subjects of fundamental stereochemical studies [1–3]; they also attract interest as key reagents in the synthesis of difficultly accessible sulfonamide derivatives, including amino acids [4, 5], amidine derivatives of amino acids [6], aminocarbonyl compounds [7], and various heterocycles such as aziridines [8, 9], benzofurans [10], and fused imidazoles [11–13].

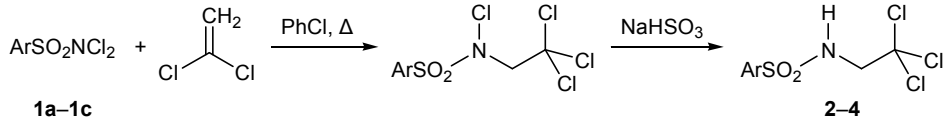
With the goal of developing methods for the synthesis of new *N*-(polychloroethyl)sulfonamides as substrates for further transformations and potential biologically active compounds, in the present work we studied reactions of some *N*-(2,2,2-trichloroethyl)-arenesulfonamides with halogen-containing electrophiles. Reactions involving the NH group of *N*-(polychloroethyl)sulfonamides have been poorly studied; a few known examples include intramolecular heterocyclizations [8, 9, 14, 15] and reactions with methyl vinyl ketone [16] and propyl, allyl, and propargyl bromides [17].

In this work we have optimized conditions of the reaction of *N*,*N*-dichloroarenesulfonamides **1a**–**1c** with 1,1-dichloroethene, leading to *N*-(2,2,2-trichloroethyl)-arenesulfonamides **2**–**4** (Scheme 1). The reaction of *N*,*N*,4-trichlorobenzenesulfonamide with 1,1-dichloroethene was reported for the first time in [18]; however, the yield of **4** was not given. We succeeded in obtaining *N*-(2,2,2-trichloroethyl)arenesulfonamides **2**–**4** in up to 92% yield via a one-pot reaction. Heating of the reactants for 6 h in chlorobenzene and the subsequent reduction of intermediate *N*-chloro amides without their isolation in the pure state favored increased yield of the target products (Scheme 1). Addition of radical initiators (AIBN or benzoyl peroxide) in the first stage considerably accelerated the process, but the yield of **2**–**4** decreased due to side reactions.

The structure of **2**–**4** was unambiguously proved by spectral and analytical data. The IR spectra of **2**–**4** characteristically displayed absorption bands due to stretching vibrations of NH (3300 cm^{-1}) and SO_2 groups (1160, 1370 cm^{-1}). In the ^1H NMR spectra of **2**–**4** we observed signals of aromatic protons, a doublet at δ ~4 ppm due to methylene protons, and a downfield triplet at ~9.0 ppm due to NH proton with the intensity ratio corresponding to the assigned structure.

Compounds **2**–**4** were brought into reactions with halogen-containing electrophiles; as a result, *N*-alkyl derivatives **5**–**7** were isolated (Scheme 2). The best

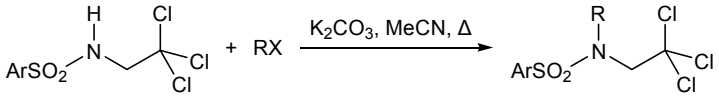
Scheme 1.



1a–1c **2–4**

1a, **2**, Ar = Ph; **1b**, **3**, R = 4-MeC_6H_4; **1c**, **4**, R = 4-ClC_6H_4.

Scheme 2.



2–4 **5**, **6a**, **6b**, **7a–7f**

X = I, Br, Cl; **5**, Ar = Ph, R = Pr; **6**, Ar = 4-MeC_6H_4, R = Et (**a**), Pr (**b**); **7**, Ar = 4-ClC_6H_4, R = Et (**a**), Pr (**b**), Bu (**c**), CH_2=$CHCH_2$ (**d**), $NCCH_2$ (**e**), $PhCH_2$ (**f**).

yields of **5**–**7** were obtained by heating the reactants in DMF or acetonitrile in the presence of an alkali metal carbonate. However, the use of DMF complicated the purification stage, thus making the procedure more laborious.

Under the given conditions, primary alkyl halides, allyl halides, chloroacetonitrile, and benzyl chloride reacted with sulfonamides **2**–**4**, whereas secondary alkyl halides failed to react, presumably for steric reasons.

The structure of **5**–**7** was confirmed by spectral data and elemental analyses. Compounds **5**–**7** showed in the IR spectra absorption bands due to stretching vibrations of SO_2 group, while no NH stretching band typical of initial sulfonamides **2**–**4** was observed. The ^{1}H and ^{13}C NMR spectra of **5**–**7** contained signals of protons and carbon nuclei of the aromatic ring, alkyl or allyl substituent, and trichloroethyl group. Methylene protons of the trichloroethyl group gave a two-proton singlet in the ^{1}H NMR spectra, in keeping with the presumed structure.

Compounds **5**–**7** were isolated as colorless crystalline solids soluble in organic solvents and insoluble in water. The absence of highly polar NH group increases the solubility of **5**–**7** in nonpolar solvents as compared to initial sulfonamides **2**–**4**.

Molecules **5**–**7** contain a trichloromethyl group and a double C=C bond (**7d**), which makes them promising as intermediate products in fine organic synthesis and obvious precursors to synthetic amino acids and functionalized acyclic and heterocyclic compounds.

EXPERIMENTAL

The IR spectra were recorded in KBr on a Bruker IFS-25 spectrometer. The ^{1}H and ^{13}C NMR spectra were measured on a Bruker DPX-400 instrument at 400.61 and 100.13 MHz, respectively, using tetramethylsilane as internal standard. The elemental analyses were obtained on a Thermo Scientific Flash EA1112 CHNS-O/MAS 200 analyzer.

***N*-(2,2,2-Trichloroethyl)benzenesulfonamide (2).** *N*,*N*-Dichloroamide **1a**, 2.49 g (11 mmol), was added in portions to a solution of 0.97 g (10 mmol) of freshly distilled 1,1-dichloroethene in 10 mL of chlorobenzene while continuously bubbling argon through the reaction mixture. The mixture was stirred for 4 h at 100°C and cooled to room temperature, a solution of 1.14 g (11 mmol) of sodium hydrogen sulfite in 30 mL of water was added, the mixture was stirred for 1 h, and the precipitate was filtered off and dried. Yield 2.42 g (85%), mp 174–175°C; published data [19]: mp 175–177°C. IR spectrum, ν, cm^{-1}: 3284 (NH), 1313, 1161 (SO_2). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.91 d (2H, CH_2, ^3J = 6.6 Hz), 7.62 m and 7.87 m (5H, C_6H_5), 8.93 t (1H, NH, ^3J = 6.6 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 60.74 (CH_2), 99.01 (CCl_3); 127.87, 130.54, 133.98, 141.23 (C_6H_4).

Compounds **3** and **4** were synthesized in a similar way.

4-Methyl-*N*-(2,2,2-trichloroethyl)benzenesulfonamide (3) was synthesized from 2.64 g (11 mmol) of *N*,*N*-dichloro amide **1b**. Yield 2.43 g (73%), mp 140–142°C; published data [20]: mp 142°C. IR spectrum, ν, cm^{-1}: 3295 (NH), 1354, 1167 (SO_2). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 3.86 d (2H, CH_2, ^3J = 6.3 Hz), 7.40 and 7.76 (4H, *AA′BB′*, C_6H_4), 8.84 t (1H, NH, ^3J = 6.3 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 60.09 (CH_2), 98.04 (CCl_3); 128.12, 129.43, 137.65, 140.68 (C_6H_5).

4-Chloro-*N*-(2,2,2-trichloroethyl)benzenesulfonamide (4) was synthesized from 2.87 g (11 mmol) of *N*,*N*-dichloro amide **1c**. Yield 2.97 g (92%), mp 178–179°C; published data [18]: mp 179°C. IR spectrum, ν, cm^{-1}: 2976 (NH), 1132, 1164 (SO_2). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 3.95 d (2H, CH_2, ^3J = 6.7 Hz), 7.68 and 7.89 (4H, *AA′BB′*, C_6H_4), 9.02 t (1H, NH, ^3J = 6.7 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 60.99 (CH_2), 98.79 (CCl_3); 128.92, 129.84, 138.04, 140.65 (C_6H_4).

***N*-Propyl-*N*-(2,2,2-trichloroethyl)benzenesulfonamide (5).** A mixture of 1.00 g (3.5 mmol) of compound **2**, 0.71 g (4.2 mmol) of 1-iodopropane, and

0.58 g (4.2 mmol) of calcined potassium carbonate in 10 mL of acetonitrile was stirred for 5 h at 60°C. The mixture was cooled to room temperature and filtered, the filtrate was evaporated under reduced pressure, and the residue was washed with 10% aqueous HCl and water, dried, and purified by recrystallization from hexane. Yield 0.96 g (83%), mp 47°C. IR spectrum, ν, cm^{-1}: 2976 (C–H_{aliph}), 1352, 1167 (SO_2). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.53 m, 1.40 m, and 3.15 m (7H, C_3H_7); 4.07 s (2H, CH_2), 7.53 m and 7.62 m (5H, C_6H_5). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 11.04 (CH_3), 21.10 (CH_2), 51.11 (CH_2), 64.33 (CH_2), 98.23 (CCl_3); 127.45, 129.23, 133.05, 139.81 (C_6H_5). Found, %: C 39.87; H 4.15; Cl 32.31; N 4.23; S 10.01. $C_{11}H_{14}Cl_3NO_2S$. Calculated, %: C 39.96; H 4.27; Cl 32.17; N 4.24; S 9.70.

Compounds **6b**, **7b**, **7c**, and **7e** were synthesized in a similar way.

***N*-Ethyl-4-methyl-*N*-(2,2,2-trichloroethyl)benzenesulfonamide (6a).** A mixture of 0.50 g (1.7 mmol) of compound **3**, 0.22 g (2.0 mmol) of bromoethane, and 0.28 g (2.0 mmol) of calcined potassium carbonate in 10 mL of acetonitrile was stirred for 7 h at 35°C. The mixture was then treated as described above in the synthesis of **5**. Yield 0.50 g (82%), mp 93–95°C. IR spectrum, ν, cm^{-1}: 2975 (C–H_{aliph}), 1532, 1161 (SO_2). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.09 m and 3.46 m (5H, CH_2CH_3), 2.37 s (3H, CH_3), 4.23 s (2H, CH_2), 7.26 and 7.69 (4H, *AA′BB′*, C_6H_4). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 12.61 (CH_3), 21.08 (CH_3), 43.82 (CH_2), 63.45 (CH_2), 7.85 (CCl_3); 126.68, 127.07, 136.67, 144.03 (C_6H_4). Found, %: C 39.85; H 4.21; Cl 32.39; N 4.38; S 9.92. $C_{11}H_{14}Cl_3NO_2S$. Calculated, %: C 39.96; H 4.27; Cl 32.17; N 4.24; S 9.70.

4-Methyl-*N*-propyl-*N*-(2,2,2-trichloroethyl)benzenesulfonamide (6b) was synthesized from 0.50 g (1.7 mmol) of compound **3** and 0.34 g (2.0 mmol) of 1-iodopropane in the presence of 0.27 g (2.0 mmol) of calcined potassium carbonate K_2CO_3. Yield 0.52 g (92%), mp 71–73°C. IR spectrum, ν, cm^{-1}: 2938 (C–H_{aliph}), 1347, 1153 (SO_2). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.79 m, 1.65 m, and 3.39 m (7H, C_3H_7); 2.44 s (3H, CH_3), 4.29 s (2H, CH_2), 7.39, 7.84 (4H, *AA′BB′*, C_6H_4). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 10.58 (CH_3), 20.53 (CH_3), 21.08 (CH_2), 50.67 (CH_2), 63.93 (CH_2), 97.83 (CCl_3); 127.04, 129.31, 136.89, 143.41 (C_6H_4). Found, %: C 41.75; H 4.58; Cl 31.02; N 4.18; S 9.13. $C_{12}H_{16}Cl_3NO_2S$. Calculated, %: C 41.81; H 4.68; Cl 30.86; N 4.06; S 9.30.

4-Chloro-*N*-ethyl-*N*-(2,2,2-trichloroethyl)benzenesulfonamide (7a) was synthesized as described above for amide **6a** from 0.60 g (1.9 mmol) of compound **4** and 0.25 g (2.3 mmol) of bromoethane in the presence of 0.31 g (2.3 mmol) of calcined potassium carbonate. Yield 0.63 g (95%), mp 102–103°C. IR spectrum, ν, cm^{-1}: 2968 (C–H_{aliph}), 1334, 1148 (SO_2). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 1.20 m and 3.57 m (5H, C_2H_5), 4.31 s (2H, CH_2), 7.52 and 7.82 (4H, *AA′BB′*, C_6H_4). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 13.06 (CH_3), 44.34 (CH_2), 63.65 (CH_2), 98.04 (CCl_3); 128.00, 129.00, 138.00, 139.00 (C_6H_4). Found, %: C 34.19; H 3.11; Cl 40.29; N 3.87; S 9.16. $C_{10}H_{11}Cl_4NO_2S$. Calculated, %: C 34.21; H 3.16; Cl 40.39; N 3.99; S 9.13.

4-Chloro-*N*-propyl-*N*-(2,2,2-trichloroethyl)benzenesulfonamide (7b) was synthesized from 1.00 g (3.0 mmol) of compound **4** and 0.61 g (3.6 mmol) of 1-iodopropane in the presence of 0.50 g (4.0 mmol) of calcined potassium carbonate. Yield 0.95 g (84%), mp 78°C. IR spectrum, ν, cm^{-1}: 2974 (C–H_{aliph}), 1352, 1161 (SO_2). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.81 m, 1.65 m, and 3.41 m (7H, C_3H_7); 4.32 s (2H, CH_2), 7.53 and 7.84 (4H, *AA′BB′*, C_6H_4). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 11.19 (CH_3), 21.10 (CH_2), 51.23 (CH_2), 64.22 (CH_2), 98.18 (CCl_3); 129.05, 129.64, 138.58, 139.74 (C_6H_4). Found, %: C 30.77; H 3.21; Cl 33.01; N 3.29; S 7.54. $C_{11}H_{13}Cl_4NO_2S$. Calculated, %: C 30.79; H 3.17; Cl 33.05; N 3.26; S 7.47.

***N*-Butyl-4-chloro-*N*-(2,2,2-trichloroethyl)benzenesulfonamide (7c)** was synthesized from 0.90 g (2.8 mmol) of compound **4** and 0.62 g (3.4 mmol) of 1-iodobutane in the presence of 0.47 g (3.4 mmol) of calcined potassium carbonate. Yield 0.93 g (91%), mp 71°C. IR spectrum, ν, cm^{-1}: 2947 (C–H_{aliph}), 1359, 1163 (SO_2). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.89 m, 1.23 m, 1.63 m, and 3.47 m (9H, C_4H_9), 4.33 s (2H, CH_2), 7.52 and 7.82 (4H, *AA′BB′*, C_6H_4). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 13.64 (CH_3), 19.96 (CH_2), 29.61 (CH_2), 49.30 (CH_2), 64.06 (CH_2), 98.10 (CCl_3); 128.95, 129.51, 138.42, 139.62 (C_6H_4). Found, %: C 38.08; H 3.89; Cl 39.01; N 4.74; S 8.57. $C_{12}H_{15}Cl_4NO_2S$. Calculated, %: C 38.02; H 3.99; Cl 38.84; N 3.69; S 8.46.

4-Chloro-*N*-(prop-2-en-1-yl)-*N*-(2,2,2-trichloroethyl)benzenesulfonamide (7d). A mixture of 1.40 g (4.3 mmol) of compound **4**, 0.62 g (5.2 mmol) of allyl bromide, and 0.72 g (5.2 mmol) of calcined potassium carbonate in 10 mL of acetonitrile was stirred for 6 h at 60°C. The mixture was then treated as described above in the synthesis of **5**. Yield 1.40 g (88%), mp 79–80°C. IR spectrum, ν, cm^{-1}: 1586 (C=C), 1351, 1164 (SO_2).

^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 4.18 d (2H, CH_2, ^3J = 2 Hz), 4.32 s (2H, CH_2), 5.25 m (2H, =CH_2), 5.49 m (1H, =CH), 7.51 and 7.84 (4H, *AA′BB′*, C_6H_4). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 51.18 (2H, CH_2), 62.00 (2H, CH_2), 97.80 (CCl_3), 121.29 (CH_2); 128.63, 129.05, 138.22, 139.29 (C_6H_4). Found, %: C 36.32; H 2.98; Cl 39.23; N 4.05; S 8.87. $C_{11}H_{11}Cl_4NO_2S$. Calculated, %: C 36.39; H 3.05; Cl 39.06; N 4.10; S 8.83.

4-Chloro-*N*-(cyanomethyl)-*N*-(2,2,2-trichloroethyl)benzenesulfonamide (7e) was synthesized from 0.50 g (1.5 mmol) of compound **4** and 0.14 g (1.8 mmol) of chloroacetonitrile in the presence of 0.25 g (1.8 mmol) of calcined potassium carbonate. Yield 0.51 g (94%), mp 112°C. IR spectrum, ν, cm^{-1}: 2212 (CN), 1351, 1164 (SO_2). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 4.32 s (2H, CH_2), 4.61 s (2H, CH_2), 7.59 and 7.84 (4H, *AA′BB′*, C_6H_4). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 37.95 (CH_2), 64.65 (CH_2), 97.08 (CCl_3), 113.78 (CN); 129.82, 130.75, 136.36, 144.90 (C_6H_4). Found, %: C 33.20; H 2.24; Cl 39.19; N 7.80; S 9.01. $C_{10}H_8Cl_4N_2O_2S$. Calculated, %: C 33.17; H 2.23; Cl 39.17; N 7.74; S 8.86.

***N*-Benzyl-4-chloro-*N*-(2,2,2-trichloroethyl)benzenesulfonamide (7f).** A mixture of 0.60 g (1.9 mmol) of compound **4**, 0.28 g (2.3 mmol) of benzyl chloride, and 0.30 g (2.2 mmol) of calcined potassium carbonate in 15 mL of acetonitrile was stirred for 5 h at 70°C. The mixture was then treated as described above in the synthesis of **5**. Yield 0.68 g (91%), mp 96°C. IR spectrum, ν, cm^{-1}: 1350, 1168 (SO_2). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 4.31 s (2H, CH_2), 4.84 s (2H, CH_2), 7.25 m (3H, C_6H_5), 7.32 m (2H, C_6H_5), 7.54 and 7.82 (4H, *AA′BB′*, C_6H_4). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 52.56 (CH_2), 62.73 (CH_2), 98.08 (CCl_3); 128.09, 129.25, 129.65, 134.01 (C_6H_5); 128.95, 129.25, 135.32, 139.03 (C_6H_4). Found, %: C 43.54; H 3.09; Cl 34.29; N 3.32; S 7.79. $C_{15}H_{13}Cl_4NO_2S$. Calculated, %: C 43.61; H 3.17; Cl 34.32; N 3.39; S 7.76.

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