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Functionalization of Highly Electrophilic *N*-(2,2,2-Trichloroethylidene)- and *N*-(2,2-Dichloro-2-phenylethylidene)arenesulfonamides with Dithiooxamide

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Abstract—Depending on the reactant ratio, dithiooxamide (ethanedithioamide) reacted as N-nucleophile or N,N′-binucleophile with highly electrophilic aldimines, *N*-(2,2,2-trichloroethylidene)- and *N*-(2,2-dichloro-2-phenylethylidene)arenesulfonamides, to give the corresponding mono- or bis-adducts, *N*-[2-polychloro-1-(arylsulfonylamino)ethyl]ethanedithioamides or *N*,*N*′-bis[2-polychloro-1-(arylsulfonylamino)ethyl]ethanedithioamides, in good yield.

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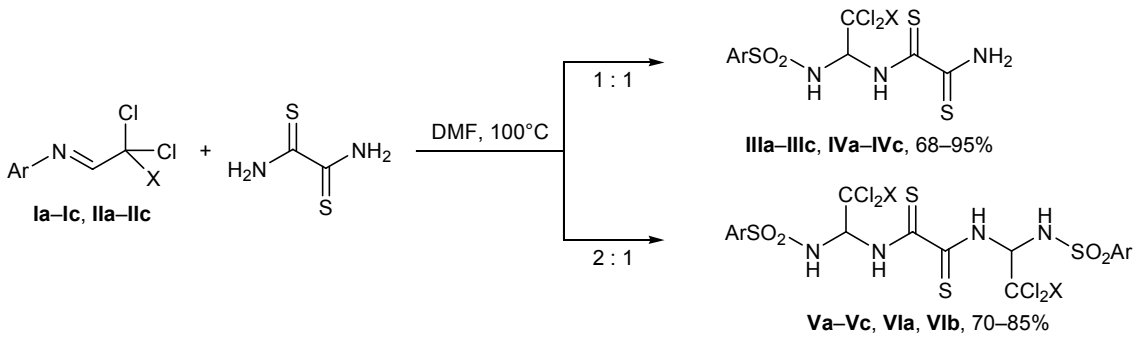
Reactions of highly electrophilic *N*-sulfonyl polychloroaldehyde imines with oxygen-, nitrogen-, sulfur-, and carbon-centered nucleophiles underlie preparatively efficient approaches to a broad series of *N*-polyhaloalkyl sulfonamides. The latter possess various functional groups, including reactive NH group and polyhalomethyl fragments, and are used in the synthesis of difficultly accessible acyclic and heterocyclic sulfonamides [1–7]. We previously showed [8] that addition of thioamides to *N*-(polychloroethylidene)-arenesulfonamides leads to the formation of polyfunctional adducts capable of undergoing cyclization to difficultly accessible 4-sulfonylaminothiazoles [6, 7]. In continuation of these studies, in the present work we examined reactions of *N*-(2,2,2-trichloroethylidene)- and *N*-(2,2-dichloro-2-phenylethylidene)arenesulfonamides **I** and **II** with dithiooxamide with a view to develop methods for the preparation of functionalized *N*-haloalkyl amides as substrates for subsequent heterocyclization and biological screening.

Dithiooxamide derivatives attract interest as starting compounds for the synthesis of a number of S,N-heterocycles [9, 10]. In addition, dithiooxamide and its derivatives are used as ligands to obtain metal complexes [11–13] and catalysts in asymmetric syntheses [11], as well as in the preparation of inorganic nanotubes [13]. Structural studies on dithiooxamide and its derivatives performed by spectral and quantum-chemical methods [14–16] are important from the theoretical viewpoint.

Scheme 1.



I, **III**, **V**, X = Cl; **II**, **IV**, **VI**, X = Ph; Ar = Ph (**a**), 4-MeC_6H_4 (**b**), 4-ClC_6H_4 (**c**).

Initial *N*-sulfonyl imines **I** and **II** are readily available via previously developed procedure which is based on radical addition of trichloroethylene and phenylacetylene to *N*,*N*-dichloroarenesulfonamides [1, 17, 18]. In reactions with imines **I** and **II** dithiooxamide acts as nitrogen-centered nucleophile which adds at the activated C=N bond. The reactions occurred most effectively on heating in dimethylformamide which ensured homogeneous process at a required temperature and the best yield of compounds **III**–**VI**. However, heating of the reaction mixture above 120°C resulted in reduced yield of adducts **III**–**VI** because of tarring.

The reaction with equimolar amounts of the reactants selectively involved only one NH_2 group of dithiooxamide with formation of the corresponding monoadducts, *N*-[2-polychloro-1-(arylsulfonylamino)-ethyl]ethanedithioamides **IIIa**–**IIIc** and **IVa**–**IVc**, in good yield. When 2 equiv of imine **I** or **II** was used, we obtained *N*,*N*′-bis[2-polychloro-1-(arylsulfonylamino)ethyl]ethanedithioamides **Va**–**Vc**, **VIa**, and **VIb** as a result of addition at both NH_2 groups of dithiooxamide. Neither S- nor N,S-addition products were formed.

The above reactions required a fairly long time (up to 15 h), i.e., dithioxamide is less reactive toward imines **I** and **II** as compared to thiourea and thioacetamide [8] which reacted with the same substrates at a higher rate. Presumably, the reactivity of dithiooxamide is reduced due to mutual electron-withdrawing effect of two thioamide groups. *N*-(Trichloroethylidene) derivatives **I** turned out to be more reactive than *N*-(2,2-dichloro-2-phenylethylidene) analogs **II**, which is consistent with stronger electron-acceptor effect of trichloromethyl group compared to dichloro(phenyl)-methyl. No appreciable effect of the substituent in the benzene ring was observed.

Compounds **III**–**VI** were isolated as orange powders which were soluble in DMSO, DMF, and aqueous alkali, poorly soluble in acetone, and insoluble in water. The structure of **III**–**VI** was unambiguously proved by their IR and NMR spectra and elemental analyses. Protons in the NH–CH–NH fragment characteristically appeared in the ^{1}H NMR spectra as three groups of signals: a doublet of doublets at δ 6.21–6.50 ppm (CH) and two downfield doublets (NH) with a coupling constant of 9.2–9.8 Hz. The spectra of monoadducts **III** and **IV** contained a broadened singlet from the NH_2 group, whereas no such signal was present in the spectra of bis-adducts **V** and **VI**. Signals from aromatic protons were also observed in the ^{1}H NMR spectra of **III**–**VI**, and the relative intensities of all signals conformed to the assumed structure.

To conclude, we have found conditions ensuring selective addition of ethanedithioamide at the C=N bond of *N*-(2,2,2-trichloroethylidene)- and *N*-(2,2-dichloro-2-phenylethylidene)arenesulfonamides. Newly synthesized *N*-polychloroalkyl sulfonamides **III**–**VI** possess pharmacophoric arenesulfonamide and ethanedithioamide fragments. They may be promising as ligands and intermediate products for the synthesis of thiazole derivatives, taking into account the presence in their molecules of RSO_2NH, NHC(S), and C–Cl moieties which can be involved in subsequent heterocyclization.

EXPERIMENTAL

The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DPX-400 spectrometer at 400.61 and 100.13 MHz, respectively, using TMS as internal reference. The IR spectra were measured on a Bruker IFS-25 spectrometer from samples pelleted with KBr.

***N*-[2,2,2-Trichloro-1-(phenylsulfonylamino)-ethyl]ethanedithioamide (IIIa).** A solution of 1.44 g (0.005 mol) of compound **Ia** and 0.60 g (0.005 mol) of ethanedithioamide in 5 ml of DMF was stirred for 12 h at 90–100°C. The mixture was cooled and diluted with 150 ml of water, the precipitate was filtered off and dissolved in 100 ml of acetone, the solution was filtered, and the filtrate was poured into 100 ml of water. The precipitate was filtered off, washed with 50 ml of diethyl ether on a filter, and dried under reduced pressure. Yield 1.65 g (81%), mp 130–132°C. IR spectrum, ν, cm^{-1}: 3427, 3321, 3243, 3148 (NH), 1610, 1341, 1166 (SO_2). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 6.47 d.d (1H, CH, ^3J = 9.6, 9.8 Hz); 7.48 m, 7.59 m, and 7.79 m (5H, C_6H_5), 9.53 d (1H, SO_2NH, ^3J = 9.6 Hz), 9.59 br.s and 10.61 br.s (1H each, NH_2), 10.57 d [1H, NHC(S), ^3J = 9.8 Hz]. ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 74.24 (CH), 99.51 (CCl_3); 126.78, 129.12, 133.07, 140.16 (C_6H_5); 188.01 (H_2NC=S), 188.42 (NHC=S). Found, %: C 29.41; H 2.39; Cl 26.01; N 10.14; S 23.48. $C_{10}H_{10}Cl_3N_3O_2S_3$. Calculated, %: C 29.53; H 2.48; Cl 26.15; N 10.33; S 23.65.

Compounds **IIIb** and **IIIc** were synthesized in a similar way.

***N*-[2,2,2-Trichloro-1-(4-chlorophenylsulfonylamino)ethyl]ethanedithioamide (IIIb)** was synthesized from 1.61 g (0.005 mol) of **Ib**. Yield 1.69 g (77%), mp 134–136°C. IR spectrum, ν, cm^{-1}: 3367, 3294, 3214, 3140 (NH), 1658, 1349, 1168 (SO_2).

^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 6.46 d.d (1H, CH, ^3J = 9.6, 9.8 Hz), 7.44 and 7.72 (4H, C_6H_4, *AA′BB′*), 9.58 d (1H, SO_2NH, ^3J = 9.6 Hz), 9.62 br.s and 10.65 br.s (1H each, NH_2), 10.58 d [1H, NHC(S), ^3J = 9.8 Hz]. ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 74.04 (CH), 99.00 (CCl_3); 128.88, 129.17, 138.19, 138.50 (C_6H_4); 185.40 (H_2NC=S), 186.77 (HNC=S). Found, %: C 27.35; H 2.15; Cl 31.65; N 9.37; S 21.97. $C_{10}H_9Cl_4N_3O_2S_3$. Calculated, %: C 27.22; H 2.06; Cl 32.14; N 9.52; S 21.80.

***N*-[2,2,2-Trichloro-1-(4-methylphenylsulfonylamino)ethyl]ethanedithioamide (IIIc)** was synthesized from 1.51 g (0.005 mol) of **Ic**. Yield 1.42 g (68%), mp 140–142°C. IR spectrum, ν, cm^{-1}: 3360, 3294, 3250, 3162 (NH), 2945, 2921 (C–H_{alk}), 1595, 1338, 1166 (SO_2). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.23 s (3H, CH_3), 6.46 d.d (1H, CH, ^3J = 9.4, 9.6 Hz), 7.18 and 7.56 (4H, C_6H_4, *AA′BB′*), 9.55 d (1H, SO_2NH, ^3J = 9.4 Hz), 9.60 br.s and 10.64 br.s (1H each, NH_2), 10.59 d (1H, NHC=S, ^3J = 9.6 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 21.1 (CH_3), 74.3 (CH), 99.4 (CCl_3); 126.2, 129.2, 135.8, 143.5 (C_6H_4); 187.7 (H_2NC=S), 188.2 (HNC=S). Found, %: C 31.47; H 2.71; Cl 25.43; N 9.67; S 22.98. $C_{11}H_{12}Cl_3N_3O_2S_3$. Calculated, %: C 31.40; H 2.87; Cl 25.28; N 9.99; S 22.86.

***N*-[2,2-Dichloro-2-phenyl-1-(phenylsulfonylamino)ethyl]ethanedithioamide (IVa).** A solution of 1.64 g (0.005 mol) of imine **IIa** and 0.60 g (0.005 mol) of ethanedithioamide in 5 ml of DMF was stirred for 15 h at 100°C, and the mixture was then treated as described above for **IIIa**. Yield 1.74 g (78%), mp 100–102°C. IR spectrum, ν, cm^{-1}: 3285, 3123 (NH), 1649, 1342, 1169 (SO_2). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 6.47 d.d (1H, CH, ^3J = 9.4, 9.7 Hz); 7.47 m, 7.59 m, 7.60 m, and 7.79 m (10H, C_6H_5); 9.54 d (1H, SO_2NH, ^3J = 9.7 Hz), 9.57 br.s and 10.08 br.s (1H each, NH_2), 10.56 d (1H, NHC=S, ^3J = 9.4 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 72.5 (CH), 93.8 (CCl_2); 125.1, 127.4, 128.1, 128.6, 130.0, 132.1, 138.9, 139.2 (C_6H_5); 186.9 (H_2NC=S), 187.6 (HNC=S). Found, %: C 42.69; H 3.22; Cl 15.73; N 9.21; S 21.38. $C_{16}H_{15}Cl_2N_3O_2S_3$. Calculated, %: C 42.86; H 3.37; Cl 15.81; N 9.37; S 21.45.

Compounds **IVb** and **IVc** were synthesized in a similar way.

***N*-[2,2-Dichloro-1-(4-chlorophenylsulfonylamino)-2-phenylethyl]ethanedithioamide (IVb)** was synthesized from 1.82 g (0.005 mol) of **IIb**. Yield 2.30 g (95%), mp 149–152°C. IR spectrum, ν, cm^{-1}: 3245 (NH), 1583, 1571, 1345, 1169 (SO_2). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 6.50 d.d (1H, CH, ^3J = 9.6, 9.8 Hz), 7.42 m and 7.67 m (5H, C_6H_5), 7.48 and 7.69 (4H, C_6H_4, *AA′BB′*), 9.21 d (1H, SO_2NH, ^3J = 9.8 Hz), 9.48 br.s and 10.61 br.s (1H each, NH_2), 10.46 d (1H, NHC=S, ^3J = 9.6 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 72.7 (CH), 93.6 (CCl_2); 127.4, 128.3, 129.9, 139.1 (C_6H_5); 128.7, 128.9, 137.2, 137.7 (C_6H_4); 186.9 (H_2NC=S), 187.6 (HNC=S). Found, %: C 39.74; H 2.86; Cl 21.90; N 8.56; S 19.88. $C_{16}H_{14}Cl_3N_3O_2S_3$. Calculated, %: C 39.80; H 2.92; Cl 22.03; N 8.70; S 19.92.

***N*-[2,2-Dichloro-1-(4-methylphenylsulfonylamino)-2-phenylethyl]ethanedithioamide (IVc)** was synthesized from 1.71 g (0.005 mol) of **IIc**. Yield 2.03 g (88%), mp 143–145°C. IR spectrum, ν, cm^{-1}: 3242 (NH), 1596, 1328, 1163 (SO_2). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.26 s (3H, CH_3), 6.48 d.d (1H, CH, ^3J = 9.2, 9.6 Hz), 7.14 and 7.52 (4H, C_6H_4, *AA′BB′*), 7.48 m and 7.67 m (5H, C_6H_5), 9.44 d (1H, SO_2NH, ^3J = 9.2 Hz), 9.47 br.s and 10.60 br.s (1H each, NH_2), 10.54 d (1H, NHC=S, ^3J = 9.6 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 21.5 (CH_3), 72.6 (CH), 93.7 (CCl_2); 126.8, 128.9, 136.1, 144.1 (C_6H_4); 127.4, 128.5, 129.8, 139.1 (C_6H_5); 187.0 (H_2NC=S), 187.4 (HNC=S). Found, %: C 44.08; H 3.65; Cl 15.17; N 8.94; S 20.68. $C_{17}H_{17}Cl_2N_3O_2S_3$. Calculated, %: C 44.15; H 3.71; Cl 15.33; N 9.09; S 20.8.

Compounds **Va**– **Vc** were synthesized as described above for **IIIa**–**IIIc** using 2 equiv of imines **Ia**–**Ic**.

***N*,*N′*-Bis[2,2,2-trichloro-1-(phenylsulfonylamino)ethyl]ethanedithioamide (Va)** was synthesized from 1.44 g (0.005 mol) of **Ia** and 0.30 g (0.0025 mol) of ethanedithioamide. Yield 1.32 g (76%), mp 128–130°C. IR spectrum, ν, cm^{-1}: 3358, 3261 (NH), 1598, 1306, 1161 (SO_2). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 6.36 d.d (2H, CH, ^3J = 9.4, 9.6 Hz); 7.48 m, 7.58 m, and 7.77 m (10H, C_6H_5); 9.34 d (2H, SO_2NH, ^3J = 9.4 Hz), 10.12 d (2H, NHC=S, ^3J = 9.6 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 74.2 (CH), 99.4 (CCl_3); 125.2, 128.7, 132.6, 139.4 (C_6H_5); 184.9 (C=S). Found, %: C 31.06; H 2.19; Cl 30.53; N 8.03; S 18.33. $C_{18}H_{16}Cl_6N_4O_4S_4$. Calculated, %: C 31.18; H 2.33; Cl 30.68; N 8.08; S 18.50.

***N*,*N′*-Bis[2,2,2-trichloro-1-(4-chlorophenylsulfonylamino)ethyl]ethanedithioamide (Vb)** was synthesized from 1.61 g (0.005 mol) of **Ib** and 0.30 g (0.0025 mol) of ethanedithioamide. Yield 1.53 g (80%), mp 118–120°C. IR spectrum, ν, cm^{-1}: 3228, 3130 (NH), 1657, 1338, 1166 (SO_2). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 6.31 d.d (2H, CH, ^3J = 9.0, 9.5 Hz), 7.44 and 7.71 (8H, C_6H_4, *AA′BB′*), 9.57 d (2H,

SO_2NH, ^3J = 9.0 Hz), 10.08 d (2H, NHC=S, ^3J = 9.5 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 74.0 (CH), 98.9 (CCl_3); 128.7. 129.2, 138.2, 138.5 (C_6H_4); 185.31 (C=S). Found, %: C 28.30; H 1.72; Cl 37.10; N 7.21; S 16.76. $C_{18}H_{14}Cl_8N_4O_4S_4$. Calculated, %: C 28.37; H 1.85; Cl 37.21; N 7.35; S 16.83.

***N*,*N*′-Bis[2,2,2-trichloro-1-(4-methylphenylsulfonylamino)ethyl]ethanedithioamide (Vc)** was synthesized from 1.51 g (0.005 mol) of **Ic** and 0.30 g (0.0025 mol) of ethanedithioamide. Yield 1.26 g (70%), mp 146–148°C. IR spectrum, ν, cm^{-1}: 3259, 3085 (NH), 1655, 1333, 1165 (SO_2). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 2.25 s (6H, CH_3), 6.29 d.d (2H, CH, ^3J = 9.4, 9.5 Hz), 7.16 and 7.58 (8H, C_6H_4, *AA′BB′*), 9.43 d (2H, SO_2NH, ^3J = 9.5 Hz), 10.10 d (2H, NHC=S, ^3J = 9.4 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 21.1 (CH_3), 74.1 (CH), 99.3 (CCl_3); 126.7, 129.4, 136.9, 143.4 (C_6H_4); 185.1 (C=S). Found, %: C 33.23; H 2.73; Cl 29.35; N 7.67; S 17.68. $C_{20}H_{20}Cl_6N_4O_4S_4$. Calculated, %: C 33.30; H 2.79; Cl 29.49; N 7.77; S 17.78.

Compounds **VIa** and **VIb** were synthesized as described above for **IVa** using 2 equiv of imine **IIa** or **IIb**.

***N*,*N*′-Bis[2,2-dichloro-2-phenyl-1-(phenylsulfonylamino)ethyl]ethanedithioamide (VIa)** was synthesized from 1.64 g (0.005 mol) of **IIa** and 0.30 g (0.0025 mol) of ethanedithioamide. Yield 1.51 g (78%), mp 93–95°C. IR spectrum, ν, cm^{-1}: 3320, 3095 (NH), 1595, 1335, 1170 (SO_2). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 6.45 d.d (2H, CH, ^3J = 9.4, 9.8 Hz); 7.46 m, 7.47 m, 7.68 m, and 7.78 m (20H, C_6H_5), 9.15 d (2H, SO_2NH, ^3J = 9.4 Hz), 10.06 d (2H, NHC=S, ^3J = 9.8 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 72.5 (CH), 93.3 (CCl_2); 125.4, 128.0, 132.4, 139.1 ($SO_2C_6H_5$); 127.2, 128.4, 130.4, 139.5 (C_6H_5); 185.3 (C=S). Found, %: C 46.48; H 3.31; Cl 18.39; N 7.35; S 16.71. $C_{30}H_{26}Cl_4N_4O_4S_4$. Calculated, %: C 46.40; H 3.37; Cl 18.26; N 7.21; S 16.51.

***N*,*N*′-Bis[2,2-dichloro-1-(4-chlorophenylsulfonylamino)-2-phenylethyl]ethanedithioamide (VIb)** was synthesized from 1.82 g (0.005 mol) of **IIb** and 0.30 g (0.0025 mol) of ethanedithioamide. Yield 1.80 g (85%), mp 160–162°C. IR spectrum, ν, cm^{-1}: 3310, 3112 (NH), 1577, 1345, 1169 (SO_2). ^1H NMR spectrum (DMSO-d_6), δ, ppm: 6.48 d.d (2H, CH, ^3J = 9.3, 9.8 Hz), 7.44 and 7.62 (8H, C_6H_4, *AA′BB′*), 7.49 m and 7.66 m (10H, C_6H_5), 9.11 d (2H, SO_2NH, ^3J = 9.3 Hz), 10.08 d (2H, NHC=S, ^3J = 9.8 Hz). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 72.6 (CH), 93.4 (CCl_2); 127.4, 128.4, 130.0, 138.9 (C_6H_5); 128.5, 128.9, 136.9, 137.8 (C_6H_4); 184.7 (C=S). Found, %: C 42.47; H 2.74; Cl 25.03; N 6.51; S 15.05. $C_{30}H_{24}Cl_6N_4O_4S_4$. Calculated, %: C 42.62; H 2.86; Cl 25.16; N 6.63; S 15.17.

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