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Dedicated to Full Member of the Russian Academy of Sciences B.A. Trofimov on his 70th anniversary

Reactions of *N*-(2,2,2-Trichloroethylidene)- and *N*-(2,2-Dichloro-2-phenylethylidene)arenesulfonamides with Biuret

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Abstract—*N*-(2,2,2-Trichloroethylidene)- and *N*-(2,2-dichloro-2-phenylethylidene)arenesulfonamides react with an equimolar amount of biuret to give 1-(1-arylsulfonylamino-2,2,2-trichloroethyl)- or 1-(1-arylsulfonylamino-2,2-dichloro-2-phenylethyl)biurets. The reactions with 2 equiv of *N*-(polychloroethylidene)arenesulfonamides involve both amino groups in the biuret molecule, yielding the corresponding 1,5-bis(1-arylsulfonylamino-2,2,2-trichloroethyl)- and 1,5-bis(1-arylsulfonylamino-2,2-dichloro-2-phenylethyl)biurets.

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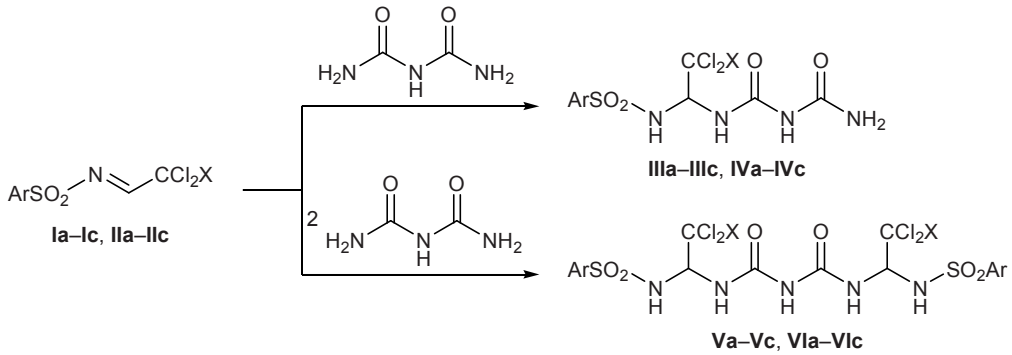
Polyamide systems having sulfonamide or urea fragments attract interest as potential ligands, strong NH acids [1], catalysts, reagents, and substrates for asymmetric synthesis and supramolecular chemistry [2]; they exhibit biological activity [3, 4] and are used in the synthesis of macrocyclic nitrogen-containing compounds [5]. Therefore, development of efficient procedures for the preparation of functionalized polyamide systems is an important problem.

Convenient synthetic approaches to various substituted amide derivatives can be based on transformations of polyhalogenated aldehyde imines. The presence of electron-withdrawing groups in molecules of such Schiff bases considerably activates the CH=N bond toward nucleophilic reagents, which is widely used for various synthetic purposes [6].

We previously studied reactions of *N*-(polychloroethylidene)arenesulfonamides with amides [7, 8], thioamides, urea, and phenylurea [9–11] with a view to develop procedures for selective synthesis of new functionalized haloalkylamide systems. While continuing studies in this line, in the present work we examined reactions of *N*-(2,2,2-trichloroethylidene)- and *N*-(2,2-dichloro-2-phenylethylidene)arensulfonamides **Ia**–**Ic** and **IIa**–**IIc** with biuret. These reactions were expected to produce polyamide systems having sulfonamide, urea, and polyhaloalkyl fragments, which may be interesting as potential biologically active sub-

Scheme 1.



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

stances, ligands, and intermediate products for subsequent transformations.

Initial Schiff bases **Ia**–**Ic** and **IIa**–**IIc** were synthesized according to the procedures developed previously, i.e., by radical reaction of the corresponding *N*,*N*-dichloroarenesulfonamides with trichloroethylene or phenylacetylene [12, 13]. Biuret reacted with Schiff bases **Ia**–**Ic** and **IIa**–**IIc** on heating the reactants to 90–100°C. The use of dimethylformamide as solvent considerably improves the yield of the addition products (the reaction mixture is homogeneous). The reactions of biuret with equimolar amounts of Schiff bases **I** and **II** involved only one amino group in the former, and the products were the corresponding 1-(1-arylsulfonylamino-2,2,2-trichloroethyl)- and 1-(1-arylsulfonylamino-2,2-dichloro-2-phenylethyl)biurets **IIIa**–**IIIc** and **IVa**–**IVc** (Scheme 1). By reactions of biuret with 2 equiv of compounds **I** and **II** we obtained 1,5-bis(1-arylsulfonylamino-2,2,2-trichloroethyl)- and 1,5-bis(1-arylsulfonylamino-2,2-dichloro-2-phenylethyl)biurets **V** and **VI**, respectively, as a result of condensation involving both NH_2 groups in the nucleophilic reagent.

Trichloroacetaldehyde derivatives **Ia**–**Ic** turned out to be more reactive in the examined process than the corresponding dichloro(phenyl)acetaldehyde derivatives **IIa**–**IIc** due to stronger electron-withdrawing effect of the trichloromethyl group as compared to dichloro(phenyl)methyl. The reaction of biuret with compounds **I** required approximately twice as short time (12 h) as analogous transformation of *N*-[2,2-dichloro-2-phenylethylidene)arenesulfonamides. The yield of addition products **III**–**VI** were 79–97%.

The structure of compounds **III**–**VI** was proved by the spectral data and elemental analyses. The IR spectra of new biuret derivatives **III**–**VI** contained absorption bands typical of NH, carbonyl, and sulfonamide groups. Reduced stretching vibration frequency of the NH and NH_2 groups indicates that they are involved in hydrogen bonding. Taking into account that molecules **III**–**VI** possess several proton-donor and proton-acceptor groups and that internal rotation about the formally single (O)C–N amide bonds is restricted, both inter- and intramolecular hydrogen bonds may be formed.

In the ^{1}H NMR spectra of addition products **III**–**VI** in DMSO-d_6, protons in the NHCHNH fragment resonated as a doublet of doublets at δ 5.7–5.9 ppm (CH) and a doublet and a singlet from the NH protons in a weaker field (δ 8.6–9.3 ppm). Presumably, the NH group in the urea fragment is involved in tautomeric transformations; therefore, the NH signal appears as a broadened singlet despite spin–spin coupling with the methine proton. In addition, the spectra contained broadened singlets due to protons in the (O)CNHC(O) and NH_2 groups and signals from aromatic protons. The chemical shifts of the NCHN proton in bis-sulfonamides **Va**–**Vc** and **VIa**–**VIc** range from δ 5.0 to 5.5 ppm [12,13]. The downfield shift of the $CHCCl_2X$ proton signal in the ^{1}H NMR spectra of biuret derivatives **III**–**VI** is likely to result from deshielding effect of the carbonyl group. Compounds **III**–**VI** displayed in the ^{13}C NMR spectra signals corresponding to carbon atoms in the carbonyl and polyhalomethyl groups, NCN carbon atom, and carbon atoms in the aromatic rings.

Substituted biurets **III**–**VI** are colorless crystalline substances which are readily soluble in DMSO, acetone, and aqueous alkali and insoluble in water. The presence of NHC=O and C–Cl fragments in their molecules makes them promising as reagents for the synthesis of nitrogen-containing heterocyclic compounds and complexes with metal salts.

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 DMSO-d_6 as solvent and tetramethylsilane as internal reference. *N*-(2,2,2-Trichloroethylidene)arenesulfonamides **Ia**–**Ic** [12] and *N*-(2,2-dichloro-2-phenylethylidene)arenesulfonamides **IIa**–**IIc** [13] were prepared according to the procedures reported previously.

1-(2,2,2-Trichloro-1-phenylsulfonylaminoethyl)-biuret (IIIa). A mixture of 2.87 g (0.01 mol) of Schiff base **Ia**, 1.03 g (0.01 mol) of biuret, and 5 ml of anhydrous dimethylformamide was stirred for 12 h at 90–100°C. The mixture was poured into 30–40 ml of water, and the precipitate was filtered off, dried, and washed with diethyl ether (30–40 ml) until colorless washings. Yield 3.08 g (79%), mp 220–222°C (decomp.). IR spectrum, ν, cm^{-1}: 1165, 1330 (SO_2); 1690, 1714 (C=O); 3025–3235 br, 3380, 3480 (NH). ^{1}H NMR spectrum, δ, ppm: 5.85 d.d (1H, CH, ^3J = 8.5 Hz); 6.80 br.s (2H, NH_2); 7.51 m, 7.63 m, and 7.82 m (5H, C_6H_5); 8.68 br.s (1H, CONHCO); 8.79 s (1H, CON**H**CH); 9.24 d (1H, SO_2NH, ^3J = 8.5 Hz). ^{13}C NMR spectrum, δ_C, ppm: 70.44 (CH); 100.99 (CCl_3); 126.67, 128.86, 132.75, 140.73 (C_{arom}); 153.19 ($CONH_2$); 155.05 (NHCONH). Found, %: C 30.95; H 2.67; Cl 27.45; N 14.25; S 8.15. $C_{10}H_{11}Cl_3N_4O_4S$. Calculated, %: C 30.83; H 2.85; Cl 27.30; N 14.38; S 8.23.

1-[2,2,2-Trichloro-1-(4-chlorophenylsulfonylamino)ethyl]biuret (IIIb) was synthesized in a similar way from 3.21 g (0.01 mol) of sulfonamide **Ib**. Yield 3.73 g (88%), mp 245–247°C (decomp.). IR spectrum, ν, cm^{-1}: 1150, 1340 (SO_2); 1700, 1720 (C=O); 3140, 3210, 3270, 3340, 3445 (NH). ^1H NMR spectrum, δ, ppm: 5.82 d.d (1H, CH, ^3J = 9.5 Hz), 6.64 br.s (2H, NH_2), 7.59 and 7.82 (4H, C_6H_4, *AA′BB′* system), 8.69 br.s (1H, CONHCO), 8.81 s (1H, CON**H**CH), 9.29 d (1H, SO_2NH, ^3J = 9.5 Hz). ^{13}C NMR spectrum, δ_C, ppm: 70.35 (CH); 100.69 (CCl_3); 128.61, 128.86, 137.65, 139.44 (C_{arom}); 153.06 ($CONH_2$); 154.96 (NHCONH). Found, %: C 28.73; H 2.41; Cl 33.86; N 13.42; S 7.44. $C_{10}H_{10}Cl_4N_4O_4S$. Calculated, %: C 28.32; H 2.38; Cl 33.44; N 13.21; S 7.56.

1-[2,2,2-Trichloro-1-(4-methylphenylsulfonylamino)ethyl]biuret (IIIc) was synthesized in a similar way from 3.01 g (0.01 mol) of sulfonamide **Ic**. Yield 3.56 g (88%), mp 225–227°C (decomp.). IR spectrum, ν, cm^{-1}: 1165, 1330 (SO_2); 1700, 1720 (C=O); 3100, 3135, 3200, 3340, 3455 (NH). ^1H NMR spectrum, δ, ppm: 2.37 s (3H, CH_3), 5.81 d.d (1H, CH, ^3J = 9.5 Hz), 6.79 br.s (2H, NH_2), 7.31 and 7.69 (4H, C_6H_4, *AA′BB′* system), 8.68 br.s (1H, CONHCO), 8.77 s (1H, CON**H**CH), 9.12 d (1H, SO_2NH, ^3J = 9.5 Hz). ^{13}C NMR spectrum, δ_C, ppm: 21.40 (CH_3); 70.79 (CH); 101.34 (CCl_3); 127.09, 129.61, 138.17, 143.45 (C_{arom}); 153.51 ($CONH_2$); 155.39 (NHCONH). Found, %: C 32.69; H 3.28; Cl 26.49; N 13.76; S 7.88. $C_{11}H_{13}Cl_3N_4O_4S$. Calculated, %: C 32.73; H 3.25; Cl 26.35; N 13.88; S 7.94.

1-(2,2-Dichloro-2-phenyl-1-phenylsulfonylaminoethyl)biuret (IVa). A mixture of 3.28 g (0.01 mol) of sulfonamide **IIa**, 1.03 g (0.01 mol) of biuret, and 5 ml of DMF was stirred for 20 h at 100°C. The mixture was then treated as described above. Yield 3.79 g (88%), mp 175–177°C. IR spectrum, ν, cm^{-1}: 1150, 1330 (SO_2); 1670, 1700 (C=O); 3275–3455 br (NH). ^1H NMR spectrum, δ, ppm: 5.95 d.d (1H, CH, ^3J = 10.0 Hz), 6.80 br.s (2H, NH_2), 7.52 m and 7.82 m (10H, C_6H_5), 8.53 br.s (1H, CONHCO), 8.59 s (1H, CON**H**CH), 8.84 d (1H, SO_2NH, ^3J = 10.0 Hz). ^{13}C NMR spectrum, δ_C, ppm: 67.33 (CH); 94.98 (CCl_2); 126.67, 128.15, 128.56, 128.86, 129.49, 136.98, 138.42, 139.73 (C_{arom}); 152.68 ($CONH_2$); 154.28 (NHCONH). Found, %: C 44.73; H 3.71; Cl 16.52; N 12.85; S 7.35. $C_{16}H_{16}Cl_2N_4O_4S$. Calculated, %: C 44.56; H 3.74; Cl 16.44; N 12.99; S 7.43.

1-[2,2-Dichloro-1-(4-chlorophenylsulfonylamino)-2-phenylethyl]biuret (IVb) was synthesized in a similar way from 3.64 g (0.01 mol) of sulfonamide **IIb**. Yield 4.25 g (91%), mp 167–169°C. IR spectrum, ν, cm^{-1}: 1150, 1340 (SO_2); 1675, 1700 (C=O); 3250–3400 br. ^1H NMR spectrum, δ, ppm: 5.96 d.d (1H, CH, ^3J = 10.3 Hz), 6.83 br.s (2H, NH_2), 7.42 m and 7.95 m (9H, C_6H_4, C_6H_5), 8.54 br.s (1H, CONHCO), 8.57 s (1H, CON**H**CH), 8.86 d (1H, SO_2NH, ^3J = 10.3 Hz). ^{13}C NMR spectrum, δ_C, ppm: 68.23 (CH); 94.60 (CCl_2); 126.97, 128.21, 128.61, 128.68, 129.58, 137.32, 138.54, 139.80 (C_{arom}); 152.87 ($CONH_2$), 155.01 (NHCONH). Found, %: C 41.38; H 3.35; Cl 22.74; N 12.12; S 6.75. $C_{16}H_{15}Cl_3N_4O_4S$. Calculated, %: C 41.26; H 3.25; Cl 22.84; N 12.03; S 6.88.

1-[2,2-Dichloro-1-(4-methylphenylsulfonylamino)-2-phenylethyl]biuret (IVc) was synthesized in a similar way from 3.42 g (0.01 mol) of sulfonamide **IIc**. Yield 3.65 g (82%), mp 196–198°C. IR spectrum, ν, cm^{-1}: 1160, 1325 (SO_2); 1660, 1695 (C=O); 3275–3440 br (NH). ^1H NMR spectrum, δ, ppm: 2.33 s (3H, CH_3), 5.96 d.d (1H, CH, ^3J = 9.8 Hz), 6.67 br.s (2H, NH_2), 7.22 m and 7.66 m (9H, C_6H_4, C_6H_5), 8.37 br.s (1H, CONHCO), 8.52 s (1H, CON**H**CH), 8.65 d (1H, SO_2NH, ^3J = 9.8 Hz). ^{13}C NMR spectrum, δ_C, ppm: 21.07 (CH_3); 68.27 (CH); 94.88 (CCl_2); 126.76, 127.07, 128.25, 129.09, 129.61, 138.20, 138.77, 142.76 (C_{arom}); 152.99 ($CONH_2$); 155.07 (NHCONH). Found, %: C 45.79; H 3.99; Cl 15.78; N 12.67; S 7.28. $C_{17}H_{18}Cl_2N_4O_4S$. Calculated, %: C 45.85; H 4.07; Cl 15.92; N 12.58; S 7.20.

1,5-Bis(2,2,2-trichloro-1-phenylsulfonylaminoethyl)biuret (Va). A mixture of 2.87 g (0.01 mol) of sulfonamide **Ia**, 0.52 g (0.005 mol) of biuret, and 5 ml of DMF was stirred for 12 h at 90–100°C. The mixture was then treated as described above. Yield 2.85 g (84%), mp 247–249°C. IR spectrum, ν, cm^{-1}: 1170, 1345 (SO_2); 1655, 1690–1715 (C=O); 3250 br (NH). ^1H NMR spectrum, δ, ppm: 5.71 d (2H, CH, ^3J = 9.6 Hz), 7.40 m and 7.94 m (10H, C_6H_5), 8.89 s (1H, CONHCO), 9.23 d (2H, SO_2NH, ^3J = 9.6 Hz). ^{13}C NMR spectrum, δ_C, ppm: 70.40 (CH); 100.69 (CCl_3); 126.68, 128.88, 132.91, 140.39 (C_{arom}); 162.50 (C=O). Found, %: C 32.48; H 2.56; Cl 31.68; N 10.78; S 9.68. $C_{18}H_{17}Cl_6N_5O_6S_2$. Calculated, %: C 31.97; H 2.53; Cl 31.46; N 10.36; S 9.48.

1,5-Bis[2,2,2-trichloro-1-(4-chlorophenylsulfonylamino)ethyl]biuret (Vb) was synthesized in a similar way from 3.21 g (0.01 mol) of sulfonamide **Ib**. Yield 3.82 g (90%), mp 252–254°C (decomp.). IR spectrum, ν, cm^{-1}: 1170, 1345 (SO_2); 1670 br (C=O); 3275–3380 br (NH). ^1H NMR spectrum, δ, ppm: 5.72 d (2H, CH, ^3J = 8.0 Hz), 7.51 and 7.75 (8H, C_6H_4, *AA′BB′* system), 8.99 s (1H, CONHCO), 9.34 d (2H,

SO_2NH, ^3J = 8.0 Hz). ^{13}C NMR spectrum, δ_C, ppm: 71.57 (CH); 101.68 (CCl_3); 129.85, 130.13, 139.00, 140.50 (C_{arom}); 153.71 (C=O). Found, %: C 29.22; H 2.09; Cl 38.45; N 9.25; S 8.45. $C_{18}H_{15}Cl_8N_5O_6S_2$. Calculated, %: C 29.02; H 2.03; Cl 38.07; N 9.40; S 8.61.

1,5-Bis[2,2,2-trichloro-1-(4-methylphenylsulfonylamino)ethyl]biuret (Vc) was synthesized in a similar way from 3.01 g (0.01 mol) of sulfonamide **Ic**. Yield 3.65 g (86%), mp 236–238°C. IR spectrum, ν, cm^{-1}: 1170, 1340 (SO_2); 1680 br (C=O); 3250–3320 br (NH). ^1H NMR spectrum, δ, ppm: 2.28 s (6H, CH_3), 5.74 d (2H, CH, ^3J = 9.9 Hz), 7.23 and 7.62 (8H, C_6H_4, *AA′BB′* system), 8.88 s (1H, CONHCO), 9.05 d (2H, SO_2NH, ^3J = 9.9 Hz). ^{13}C NMR spectrum, δ_C, ppm: 20.49 (CH_3); 71.04 (CH); 100.98 (CCl_3); 127.55, 128.92, 137.68, 140.25 (C_{arom}); 153.53 (C=O). Found, %: C 34.38; H 3.09; Cl 30.28; N 9.86; S 9.25. $C_{20}H_{21}Cl_6N_5O_6S_2$. Calculated, %: C 34.11; H 3.01; Cl 30.20; N 9.94; S 9.10.

1,5-Bis(2,2-dichloro-2-phenyl-1-phenylsulfonylaminoethyl)biuret (VIa). A mixture of 3.28 g (0.01 mol) of sulfonamide **IIa**, 0.52 g (0.005 mol) of biuret, and 5 ml of DMF was stirred for 20 h at 100°C. The mixture was then treated as described above. Yield 3.50 g (92%), mp 85–87°C. IR spectrum, ν, cm^{-1}: 1160, 1340 (SO_2); 1655, 1700 (C=O); 3275–3360 br (NH). ^1H NMR spectrum, δ, ppm: 5.94 d.d (2H, CH, ^3J = 9.6, 10.2 Hz), 7.35 m and 7.95 m (20H, C_6H_5), 8.58 s (1H, CONHCO), 8.83 d (2H, CON**H**CH, ^3J = 9.6 Hz), 8.85 d (2H, SO_2NH, ^3J = 10.2 Hz). ^{13}C NMR spectrum, δ_C, ppm: 68.21 (CH); 94.43 (CCl_2); 126.83, 127.22, 127.89, 128.13, 128.20, 128.86, 128.99, 129.85 (C_{arom}); 152.79 (C=O). Found, %: C 47.56; H 3.48; Cl 18.55; N 9.25; S 8.25. $C_{30}H_{27}Cl_4N_5O_6S_2$. Calculated, %: C 47.44; H 3.58; Cl 18.67; N 9.22; S 8.44.

1,5-Bis[2,2-dichloro-1-(4-chlorophenylsulfonylamino)-2-phenylethyl]biuret (VIb) was synthesized in a similar way from 3.64 g (0.01mol) of sulfonamide **IIb**. Yield 4.04 g (97%), mp 78–80°C. IR spectrum, ν, cm^{-1}: 1170, 1340 (SO_2); 1660, 1700 (C=O); 3280–3370 br (NH). ^1H NMR spectrum, δ, ppm: 5.95 d.d (2H, CH, ^3J = 9.5, 10.0 Hz), 7.39 m and 7.95 m (18H, C_6H_4, C_6H_5), 8.58 s (1H, CONHCO), 8.83 d (2H, CON**H**CH, ^3J = 9.5 Hz), 8.86 d (2H, SO_2NH, ^3J = 10.0 Hz). ^{13}C NMR spectrum, δ_C, ppm: 69.26 (CH); 94.60 (CCl_2); 126.94, 127.33, 127.55, 127.97, 128.00, 128.76, 129.00, 129.58 (C_{arom}); 152.84 (C=O). Found, %: C 43.65; H 3.09; Cl 25.46; N 8.39; S 7.78. $C_{30}H_{25}Cl_6N_5O_6S_2$. Calculated, %: C 43.50; H 3.04; Cl 25.68; N 8.45; S 7.74.

1,5-Bis[2,2-dichloro-1-(4-methylphenylsulfonylamino)-2-phenylethyl]biuret (VIc) was synthesized in a similar way from 3.42 g (0.01 mol) of sulfonamide **IIc**. Yield 3.51 g (89%), mp 82–84°C. IR spectrum, ν, cm^{-1}: 1165, 1340 (SO_2); 1660 (C=O); 3275–3370 br (NH). ^1H NMR spectrum, δ, ppm: 2.34 s (6H, CH_3); 5.93 d.d (2H, CH, ^3J = 9.4, 10.1 Hz), 7.32 m and 7.98 m (18H, C_6H_4, C_6H_5), 8.49 s (1H, CONHCO), 8.82 d (2H, CON**H**CH, ^3J = 9.4 Hz), 8.87 d (2H, SO_2NH, ^3J = 10.1 Hz). ^{13}C NMR spectrum, δ_C, ppm: 24.73 (CH_3); 68.85 (CH); 94.58 (CCl_2); 124.99, 127.03, 127.45, 127.99, 128.08, 128.67, 129.12, 129.62 (C_{arom}); 152.96 (C=O). Found, %: C 48.85; H 3.95; Cl 18.11; N 8.95; S 8.05. $C_{32}H_{31}Cl_4N_5O_6S_2$. Calculated, %: C 48.80; H 3.97; Cl 18.01; N 8.89; S 8.14.

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