

Reaction of *N,N*-Dichlorosulfonamides with Tribromoethylene

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Abstract—Reaction of aryl- and trifluoromethanesulfonic acids *N,N*-dichloroamides with tribromoethylene led to the formation of a mixture of *N*-(2,2-dibromo-2-chloroethylidene)- and *N*-(2,2,2-tribromoethylidene)amides of the corresponding sulfonic acids. The azomethines ratio is governed by the reaction temperature.

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It was shown formerly [1] that *N,N*-dichlorobenzene-sulfonamide (**Ia**) reacted with tribromoethylene giving *N*-(2,2-dibromo-2-chloroethylidene)amide of benzene-sulfonic acid (**IIa**). Imine **IIa** was suggested [1] to exist as a mixture of *E*- and *Z*-isomers for the proton and carbon atom of the CH=N group appeared as two signals in the ¹H and ¹³C NMR spectra respectively.

In extension of the investigation of reactions between *N,N*-dichlorosulfonamides and polyhaloethenes we studied the reaction of *N,N*-dichloroamides of benzene-sulfonic acid (**Ia**), 4-chlorobenzenesulfonic acid (**Ib**), and trifluoromethanesulfonic acid (**Ic**) with tribromoethylene under various conditions. The reactions were carried out using four-fold molar excess of tribromoethylene in CCl₄ solution.

It was established that the reaction of dichloroarene-sulfonamides **Ia** and **Ib** with tribromoethylene completed in 5–8 h at boiling the reaction mixture or within 7–10 days at 15–20°C and provided a mixture of arene-sulfonylimines of dibromochloroacetic (**IIa** and **IIb**) and tribromoacetic (**IIIa** and **IIIb**) aldehydes. Therewith the molar ratio of azomethines **II:III** varied from 4:3 at boiling to 3:1 at lower temperature (Scheme 1).

The reaction of *N,N*-dichlorotrifluoromethanesulfonamide (**Ic**) with tribromoethylene started at room

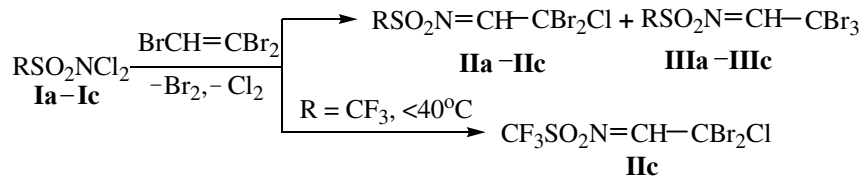
temperature under sunlight and proceeded with a considerable heat evolution. The the bright sunlight the temperature of the reaction mixture attained 100°C and more. If the reaction temperature was maintained below 40°C the process led to the exclusive formation of trifluoromethanesulfonic acid *N*-(2,2-dibromo-2-chloroethylidene)amide (**IIc**) (Scheme 1). In other cases the reaction yielded a mixture of imines **IIc** and **IIIc** in a molar ratio 1:1.

The liberated halogens added to the tribromoethylene giving a mixture of polybromochloroethanes [1].

Therefore as mentioned above the chemoselectivity of reaction between dichloroamides **Ia–Ic** and tribromoethylene depended on the temperature of the process: On decreasing the temperature the amount of tribromoacetic aldehyde sulfonylimines **III** diminished, and from compound **Ic** formed exclusively dibromochloroacetic aldehyde sulfonylimine **IIc**.

Under low temperature dichloroamides **Ia–Ic** added to tribromoethylene forming saturated adduct **C**. The possibility of adducts of type **C** formation in the reactions of *N,N*-dichlorosulfonamides with polychloroethenes under mild conditions we showed in [2]. Saturated adduct **C** is further dehalogenated (slowly at the room tempera-

Scheme 1.



R = C₆H₅ (**a**), 4-ClC₆H₄ (**b**), CF₃ (**c**).

ture and fast at heating) to give dibromochloroacetic aldehyde sulfonylimines **IIa–IIc**. Thus the stages of dichloroamide addition to tribromoethylene and dehalogenation occur in succession.

In reaction at heating saturated adduct **C** is unstable and rapidly eliminates halogen, namely, the addition and dehalogenation stages proceed simultaneously (Scheme 2). Therefore in the reaction mixture certain amounts exist of molecular chlorine, bromine, and bromine chloride which may concurrently react with radical-adduct **B** giving saturated adducts **D** and further azomethines **IIIa–IIIc**.

It should be also admitted that *N,N*-dichloroamides **Ia–Ic** may react with the liberated bromine to form *N,N*-dibromoamides that then add to tribromoethylene giving imines **IIIa–IIIc**.

The imine mixtures formation was proved by ^1H and ^{13}C NMR spectroscopy. For instance, in the ^1H NMR spectra of compounds **IIa–IIc** and **IIIa–IIIc** two signals of azomethine protons appeared in the region 8.3–8.6 ppm of integral intensity ratio from 4:3 to 3:1. The signals of aromatic protons of compound **IIb** gave rise to two groups of signals with the same ratio of integral

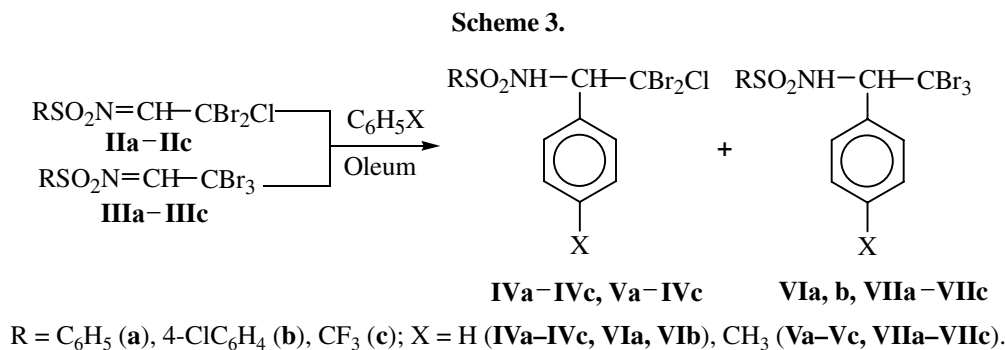
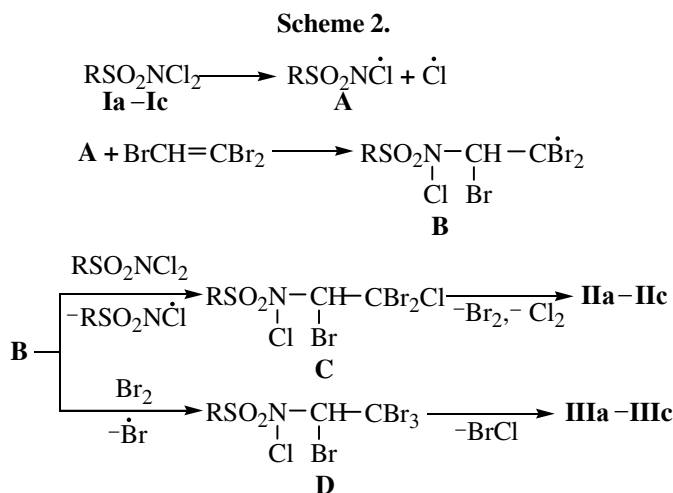
intensity as the azomethine protons. In the ^{13}C NMR spectra signals were observed of CBr_3 group at 31 ppm and CBr_2Cl group at 53 ppm, and all the other carbon atoms of the molecules (azomethine and aromatic) appeared as two groups of signals.

The presence of the mixture containing dibromochloro- and tribromoacetic aldehyde imines was also confirmed by the study of their transformation products which in contrast to imines **II** and **III** were chemically stable, easily isolable from the reaction mixture, and fit for further investigation. For instance, the C-amidoalkylating activity of the prepared polybromoaldehydes sulfonylimines was tested on reactions with benzene and toluene.

The C-amidoalkylation of arenes (Scheme 3) proceeded similarly to the previously studied reactions of chloral arenesulfonyl- and trifluoromethanesulfonylimines [3, 4] in the presence of oleum within 3–5 h and led to the formation of corresponding *N*-(1-aryl-2-polyhaloethyl)-sulfonamides **IV–VII**.

In the ^1H NMR spectra of the alkylation products of benzene (**IVa–IVc** and **VIa–VIc**) and toluene (**Va–Vc** and **VIIa–VIIc**) the fragment $\text{NH}-\text{CH}$ gave rise to two doublets from NH group and two doublets from CH group with the same ratio of the integral intensities of signals as was observed for the signals of the azomethine protons in the spectra of the initial mixtures of azomethines **II** and **III**. In the ^{13}C NMR spectra signals were observed of CBr_2Cl groups (65–68 ppm) and CBr_3 groups (46–50 ppm), and all the other carbon atoms of the molecules appeared as two groups of signals corresponding to two amides containing either dibromochloromethyl or tribromomethyl groups.

The study of the mixture produced by reaction of imines **IIc** and **IIIc** with toluene by GC-MS procedure confirmed the presence of two amidoalkylated arenes. The mass spectrum of one among them according to the molecular ion peak and those of fragment ions (see



EXPERIMENTAL) was consistent with the structure of dibromochloroethylamide **Vc**. We failed to observe the molecular ion peak of the second compound, but all the other peaks were well consistent with the structure of amide **VIIc**. In the mass spectra of both amides **IIc** and **IIIc** the most abundant were the fragment peaks [$M - CBr_2Cl$] and [$M - CBr_3$].

Thus we established for the first time that the reaction of arene- and trifluoromethanesulfonic acid dichloroamides with tribromoethylene led to the formation of a mixture of arene- or trifluoromethanesulfonylimines of bromal and dibromochloroacetic aldehyde. The dependence of the molar ratio of the azomethines on the process temperature was observed.

EXPERIMENTAL

1H , ^{13}C , ^{15}N , and ^{19}F NMR spectra were registered on a spectrometer Bruker DPX-400 at operating frequencies 400.13, 101.61, 40.56, and 376 MHz respectively from solutions in $CDCl_3$ or $DMSO-d_6$. Chemical shifts of 1H and ^{13}C were measured with respect to TMS with accuracy of 0.01 ppm, as internal references for ^{15}N and ^{19}F were used nitromethane and trichlorofluoromethane respectively. The coupling constants J_{HH} and J_{CF} were measured with accuracy of 0.1 Hz. IR spectra were recorded on a spectrophotometer Specord 75IR from KBr pellets. The mass spectrum was taken on Shimadzu GC-17/GCMS-QP5050-1 instrument.

Oleum used in reactions contained 5% of SO_3 .

Reaction of *N,N*-dichlorobenzenesulfonamide with tribromoethylene. A solution of 2.26 g (0.01 mol) of dichloroamide **Ia** in 10.6 g (0.04 mol) of tribromoethylene and 4 ml of CCl_4 was boiled for 6 h with continuous bubbling of argon. Then the reaction mixture was maintained for 10 h at $-10^\circ C$, the formed precipitate was separated by filtration, washed on the filter with a little cold CCl_4 , and dried. We obtained 3.5 g of a mixture of benzenesulfonic acid *N*-(2,2-dibromo-2-chloroethylidene)amide (**IIa**) and *N*-(2,2,2-tribromoethylidene)amide (**IIIa**) in a ratio 4:3. 1H NMR spectrum ($CDCl_3$), δ , ppm, imine **IIa**: 7.60–8.0 m (5H, C_6H_5), 8.47 s (1H, $N=CH$); imine **IIIa**: 7.60–8.0 m (5H, C_6H_5), 8.35 s (1H, $N=CH$). ^{13}C NMR spectrum, δ , ppm, imine **IIa**: 53.07 ($CBBr_2Cl$), 128.10, 129.16, 134.32, 135.83 (C_6H_5), 164.88 ($CH=N$); imine **IIIa**: 31.60 ($CBBr_3$), 128.06, 129.18, 134.28, 135.94 (C_6H_5), 165.22 ($CH=N$).

Reaction of *N,N*-dichloro(4-chlorobenzene)sulfonamide with tribromoethylene. In the same way from

2.6 g (0.01 mol) of dichloroamide **Ib** and 10.6 g (0.04 mol) tribromoethylene was obtained 4.0 g of a mixture of 4-chlorobenzenesulfonic acid *N*-(2,2-dibromo-2-chloroethylidene)amide (**IIb**) and *N*-(2,2,2-tribromoethylidene)amide (**IIIb**) in a ratio 4:3. 1H NMR spectrum ($CDCl_3$), δ , ppm, imine **IIb**: 7.56 and 7.93 AA'BB' (C_6H_4), 8.46 s (1H, $N=CH$); imine **IIIb**: 7.58 and 7.93 AA'BB' (C_6H_4), 8.34 s (1H, $N=CH$). ^{13}C NMR spectrum, δ , ppm, imine **IIb**: 53.19 ($CBBr_2Cl$), 129.95, 134.67, 141.63 (C_6H_4), 165.55 ($CH=N$); imine **IIIb**: 31.66 ($CBBr_3$), 129.91, 134.78, 141.58 (C_6H_4), 165.89 ($CH=N$).

Reaction of *N,N*-dichlorotrifluoromethanesulfonamide with tribromoethylene. Through a solution of 2.18 g (0.01 mol) of dichloroamide **Ic** in 10.6 g (0.04 mol) of tribromoethylene was bubbled argon for 5 min, then the reaction mixture was left standing at room temperature in a bright sunlight. The reaction mixture self-heated to $100^\circ C$. After the end of heat liberation the solution was kept for 23 h at room temperature. According to 1H NMR spectrum the reaction products contained a mixture of trifluoromethanesulfonic acid *N*-(2,2-dibromo-2-chloroethylidene)amide (**IIc**) and *N*-(2,2,2-tribromoethylidene)amide (**IIIc**) in a ratio 1:1. 1H NMR spectrum (tribromoethylene), δ , ppm: 8.52 s (1H, $CH=N$) (**IIIc**), 8.63 s (1H, $CH=N$) (**IIc**).

***N*-(2,2-Dibromo-2-chloroethylidene)trifluoromethanesulfonamide (IIc).** A solution of 2.18 g (0.01 mol) of dichloroamide **Ic** in 10.6 g (0.04 mol) of tribromoethylene was maintained 24 h in a sunlight under argon atmosphere at a temperature not higher than $40^\circ C$. According to 1H NMR spectrum the reaction product contained imine **IIc** without impurity of imine **IIIc**. The obtained imine was used in further synthesis without isolation. IR spectrum of the reaction mixture (microfilm), ν , cm^{-1} : 1120, 1200, 1390 (CF_3SO_2), 1620 ($C=N$). 1H NMR spectrum (tribromoethylene), δ , ppm: 8.63 s ($CH=N$).

C-Amidoalkylation of benzene with a mixture of imines IIa and IIIa. To 3.5 g of a mixture of imines **IIa** and **IIIa** obtained as described above was added 10 ml of benzene and 0.5 ml of oleum. The reaction mixture was stirred for 5 h, diluted with 20 ml of water, and the acid was neutralized with sodium carbonate. The separated precipitate was filtered off, washed with water on the filter, and dried to obtain 3.2 g of a mixture of benzenesulfonic acid *N*-(1-phenyl-2,2-dibromo-2-chloroethyl)amide (**IVa**) and *N*-(1-phenyl-2,2,2-tribromoethyl)amide (**VIa**) in a ratio 4:3 (1H NMR data). 1H NMR

spectrum (DMSO- d_6), δ , ppm, amide **IVa**: 5.19 d (1H, CH, $^3J_{\text{CH-NH}}$ 10.5 Hz), 7.0–7.6 m (10H, $2\text{C}_6\text{H}_5$), 9.14 d (1H, NH, $^3J_{\text{CH-NH}}$ 10.5 Hz); amide **VIa**: 5.20 d (1H, CH, $^3J_{\text{CH-NH}}$ 10.5 Hz), 7.0–7.6 m (10H, $2\text{C}_6\text{H}_5$), 9.09 d (1H, NH, $^3J_{\text{CH-NH}}$ 10.5 Hz). ^{13}C NMR spectrum (DMSO- d_6), δ , ppm, amide **IVa**: 68.24 (CBr_2Cl), 72.60 (CH), 126.32, 127.22, 128.35, 128.42, 129.85, 132.01, 133.97, 140.39 ($2\text{C}_6\text{H}_5$); amide **VIa**: 49.94 (CBr_3), 72.87 (CH), 126.36, 127.11, 128.26, 128.37, 129.92, 131.96, 134.20, 140.33 ($2\text{C}_6\text{H}_5$).

C-Amidoalkylation of toluene with a mixture of imines IIa and IIIa was carried out similarly using 3.5 g of a mixture of imines **IIa** and **IIIa** and 10 ml of toluene. We obtained 3.8 g of a mixture of benzenesulfonic acid *N*-(1-tolyl-2,2-dibromo-2-chloroethyl)-amide (**Va**) and *N*-(1-tolyl-2,2,2-tribromoethyl)-amide (**VIIa**) in a ratio 4:3 (^1H NMR data). ^1H NMR spectrum (DMSO- d_6), δ , ppm, amide **Va**: 2.17 s (3H, CH_3), 5.14 d (1H, CH, $^3J_{\text{CH-NH}}$ 10.5 Hz), 6.8–7.6 m (9H, $\text{C}_6\text{H}_5+\text{C}_6\text{H}_4$), 9.07 d (1H, NH, $^3J_{\text{CH-NH}}$ 10.5 Hz); amide **VIIa**: 2.16 s (3H, CH_3), 5.15 d (1H, CH, $^3J_{\text{CH-NH}}$ 10.5 Hz), 6.8–7.6 m (9H, $\text{C}_6\text{H}_5+\text{C}_6\text{H}_4$), 9.02 d (1H, NH, $^3J_{\text{CH-NH}}$ 10.5 Hz). ^{13}C NMR spectrum (DMSO- d_6), δ , ppm, amide **Va**: 20.63 (CH_3), 68.69 (CBr_2Cl), 72.46 (CH), 126.33, 127.79, 128.42, 129.73, 131.15, 131.92, 137.71, 140.53 ($\text{C}_6\text{H}_5+\text{C}_6\text{H}_4$); amide **VIIa**: 21.03 (CH_3), 50.49 (CBr_3), 72.75 (CH), 126.36, 127.69, 128.37, 129.81, 131.37, 131.86, 137.61, 140.45 ($\text{C}_6\text{H}_5+\text{C}_6\text{H}_4$).

C-Amidoalkylation of benzene with a mixture of imines IIb and IIIb was carried out similarly using 4.0 g of a mixture of imines **IIb** and **IIIb** and 12 ml of benzene. We obtained 4.1 g of a mixture of 4-chlorobenzenesulfonic acid *N*-(1-phenyl-2,2-dibromo-2-chloroethyl)-amide (**IVb**) and *N*-(1-phenyl-2,2,2-tribromoethyl)-amide (**VIb**) in a ratio 4:3 (^1H NMR data). ^1H NMR spectrum (DMSO- d_6), δ , ppm, amide **IVb**: 5.18 d (1H, CH, $^3J_{\text{CH-NH}}$ 10.8 Hz), 7.0–7.6 m (9H, $\text{C}_6\text{H}_4+\text{C}_6\text{H}_5$), 9.20 d (1H, NH, $^3J_{\text{CH-NH}}$ 10.8 Hz); amide **VIb**: 5.19 d (1H, CH, $^3J_{\text{CH-NH}}$ 10.8 Hz), 7.0–7.6 m (9H, $\text{C}_6\text{H}_4+\text{C}_6\text{H}_5$), 9.15 d (1H, NH, $^3J_{\text{CH-NH}}$ 10.8 Hz). ^{13}C NMR spectrum (DMSO- d_6), δ , ppm, amide **IVb**: 68.36 (CBr_2Cl), 73.16 (CH), 127.83, 128.87, 129.04, 130.43, 134.46, 137.53, 139.68 ($\text{C}_6\text{H}_4+\text{C}_6\text{H}_5$); amide **VIb**: 49.93 (CBr_3), 73.43 (CH), 127.73, 128.89, 128.99, 130.51, 134.70, 137.50, 139.62 ($\text{C}_6\text{H}_4+\text{C}_6\text{H}_5$).

C-Amidoalkylation of toluene with a mixture of imines IIb and IIIb was carried out similarly using 4.0 g of a mixture of imines **IIb** and **IIIb** and 12 ml of toluene. We obtained 3.9 g of a mixture of 4-chloro-

benzenesulfonic acid *N*-(1-tolyl-2,2-dibromo-2-chloroethyl)-amide (**Vb**) and *N*-(1-tolyl-2,2,2-tribromoethyl)-amide (**VIIb**) in a ratio 4:3 (^1H NMR data). ^1H NMR spectrum (DMSO- d_6), δ , ppm, amide **Vb**: 2.20 s (3H, CH_3), 5.11 d (1H, CH, $^3J_{\text{CH-NH}}$ 10.6 Hz), 6.91, 7.30, 7.52 m (8H, $2\text{C}_6\text{H}_4$), 9.10 d (1H, NH, $^3J_{\text{CH-NH}}$ 10.6 Hz); amide **VIIb**: 2.20 s (3H, CH_3), 5.11 d (1H, CH, $^3J_{\text{CH-NH}}$ 10.6 Hz), 6.90, 7.30, 7.50 m (8H, $2\text{C}_6\text{H}_4$), 9.05 d (1H, NH, $^3J_{\text{CH-NH}}$ 10.6 Hz). ^{13}C NMR spectrum (DMSO- d_6), δ , ppm, amide **Vb**: 20.69 (CH_3), 68.15 (CBr_2Cl), 72.59 (CH), 127.95, 128.46, 128.61, 129.92, 131.01, 137.07, 137.98, 139.20 ($2\text{C}_6\text{H}_4$); amide **VIIb**: 20.69 (CH_3), 49.80 (CBr_3), 72.88 (CH), 127.86, 128.49, 128.56, 130.00, 131.25, 137.03, 137.90, 139.12 ($2\text{C}_6\text{H}_4$).

C-Amidoalkylation of benzene with imine IIc. To a solution of imine **IIc** obtained as above described from 2.18 g of dichloroamide **Ic** and tribromoethylene was added 12 ml of benzene and 0.5 ml of oleum. After stirring for 3 h the solvent was removed in a vacuum, the residue was washed with water, dried, and recrystallized from hexane. Yield of trifluoromethanesulfonic acid *N*-(1-phenyl-2,2-dibromo-2-chloroethyl)-amide (**IVc**) 1.11 g (25%), mp 136–138°C. IR spectrum (KBr), cm^{-1} : 1130, 1200, 1230, 1375 (CF_3SO_2), 3275 (NH). ^1H NMR spectrum, δ , ppm: 5.29 d (1H, CH, $^3J_{\text{CH-NH}}$ 10.2 Hz), 6.39 d (1H, NH, $^3J_{\text{CH-NH}}$ 10.2 Hz), 7.35–7.55 m (5H, C_6H_5). ^{13}C NMR spectrum, δ , ppm: 65.45 (CBr_2Cl), 73.20 (CH), 119.04 q (CF_3 , $^1J_{\text{C-F}}$ 321.7 Hz), 128.44, 129.29, 130.06, 133.51 (C_6H_5).

C-Amidoalkylation of toluene with a mixture of imines IIc and IIIc was performed in the same way using a mixture of imines **IIc** and **IIIc** prepared from 2.18 g of dichloroamide **Ic** and tribromoethylene. Yield of a mixture of trifluoromethanesulfonic acid *N*-(1-tolyl-2,2-dibromo-2-chloroethyl)-amide (**Vc**) and *N*-(1-tolyl-2,2,2-tribromoethyl)-amide (**VIIc**), 1:1, 0.8 g. IR spectrum (KBr), cm^{-1} : 3250 (NH), 1420, 1370, 1220, 1180, 1120 (CF_3SO_2). ^1H NMR spectrum (CDCl_3), δ , ppm, amide **Vc**: 2.36 s (3H, CH_3), 5.24 d (1H, CH, $^3J_{\text{CH-NH}}$ 10.2 Hz), 6.17 d (1H, NH, $^3J_{\text{CH-NH}}$ 10.2 Hz), 7.20, 7.42 AA'BB' (4H, C_6H_4); amide **VIIc**: 2.36 s (3H, CH_3), 5.28 d (1H, CH, $^3J_{\text{CH-NH}}$ 10.2 Hz), 6.18 (1H, NH, $^3J_{\text{CH-NH}}$ 10.2 Hz), 7.20, 7.43 AA'BB' (4H, C_6H_4). ^{13}C NMR spectrum (CDCl_3), δ , ppm, amide **Vc**: 21.40 (CH_3), 66.05 (CBr_2Cl), 73.01 (CH), 119.01 q (CF_3 , $^1J_{\text{C-F}}$ 321.1 Hz), 129.06, 129.12, 130.77, 140.19 (C_6H_4); amide **VIIc**: 21.38 (CH_3), 46.59 (CBr_3), 73.21 (CH), 119.04 q (CF_3 , $^1J_{\text{C-F}}$ 321.0 Hz), 129.14, 129.24, 130.51, 140.22 (C_6H_4). ^{19}F NMR spectrum, δ , ppm: –76.93 s, –76.89 c. ^{15}N NMR spectrum δ , ppm: –279.4 (**Vc**), –278.5 (**VIIc**). Mass

spectrum, m/z (I_{rel} , %) amide **Vc**: 457/459/461 (0.74) $[M]^+$, 343/345 (0.99) $[M - Br - Cl]^+$, 309/311/313 (0.93) $[M - CF_3SO_2NH]^+$, 252 (100) $[M - CBr_2Cl]^+$, 205/207/209 (3.29) $[CBr_2Cl]^+$, 118 (19.1) $[M - CF_3SO_2H - CBr_2Cl]^+$, 103 (4.65) $[M - CF_3SO_2NH - CBr_2Cl - H]^+$, 91 (11.21) $[C_6H_5CH_2]^+$, 69 (14.99) $[CF_3]^+$; amide **VIc**: 423/425/427 (0.41) $[M - HBr]^+$, 343/345 (0.75) $[M - Br_2]^+$, 275/277/279 (1.27) $[M - CF_3SO_2NH - HBr]^+$, 252 (100) $[M - CBr_3]^+$, 195/197 (1.07) $[M - CF_3SO_2NH - Br_2]^+$, 196/198 (3.4) $[M - CF_3SO_2NH - Br_2 + H]^+$, 171/173/175 (5.17) $[CHBr_2]^+$, 118 (28.00) $[M - CF_3SO_2H - CBr_3]^+$, 103 (9.44) $[M - CF_3SO_2NH - CBr_3 - H]^+$, 91 (16.37) $[C_6H_5CH_2]^+$, 69 (23.22) $[CF_3]^+$.

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