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Reactions of 1-Bromo-1-nitro-3,3,3-trichloropropene with *O*- and *N*-Nucleophiles

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Abstract—Reactions of 1-bromo-1-nitro-3,3,3-trichloropropene with alcohols, benzidine and *o*-, *m*-, and *p*-phenylenediamines was investigated and the reaction conditions were developed. The alkoxylation and amination products and an unusual nitrogen-containing bis-aziridine were obtained; their structures were characterized by the spectral (IR, ^{1}H, ^{13}C NMR, HMQC, and HMBC) methods.

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Halonitroalkenes containing highly electrophilic multiple C=C bond are widely used as reactive synthons for obtaining organic compounds of various types, including nitrogen-containing carbo- and heterocycles [1].

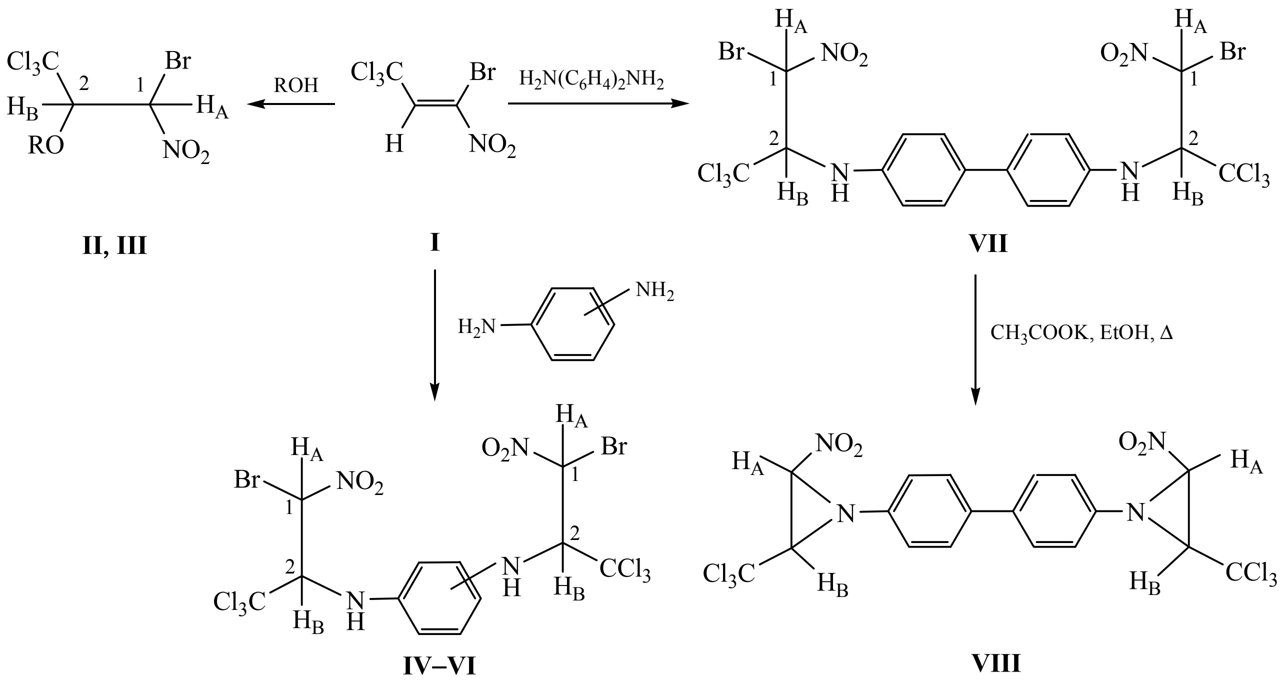
In this connection the 1-bromo-1-nitro-3,3,3-trichloropropene is of undoubted interest [2]. The chemistry of this compound was poorly studied up to now [3], meanwhile the combined presence of nitro-, trichloromethyl groups, and bromine atom in its molecule is promising for obtaining potential biologically active materials on the basis of this nitroalkene. Its nitroprecursor, 1,1,1-trichloro-3-nitropropene, is known to possess herbicide and fumigant activity [4], and its alkoxylation products exhibit spasmolitic [5] and antimicrobic [6] properties.

We examined a reaction of 1-bromo-1-nitro-3,3,3-trichloropropene **I** with some aliphatic alcohols and aromatic diamines. The alkoxylation reaction of 1-bromo-1-nitro-3,3,3-trichloropropene **I** occurs under the mild conditions at reflux in excess of the corresponding alcohol over 1.5 h to form alkoxyderivatives **II**, **III** in high yields (96%), in contrast to the same reaction of its nitro-precursor (prolonged reflux in alcohol for 1–4 days [5]). Note that compound **II** obtained earlier by another method (yield 34%) was charactirized only by its boiling point [6].

The reaction of 1-bromo-1-nitro-3,3,3-trichloropropene **I** with aromatic diamines proceeds successfully in methanol solution at room temperature within 1–3 h to give oily or crystalline bis-adducts **IV–VII** in 63–94% yield.

Structures of adducts **II–VII** were proved by IR, ^{1}H and ^{13}C NMR spectroscopy. According to the ^{1}H and ^{13}C NMR spectra containing two sets of signals, compounds **II**, **III**, **VI,** and **VII** are mixtures of two diastereomers in the ratio a:b = 2:1 (**II**, **III**, **VII**) or a:b = 3:1 (**VI**). We assign the upfield signals H_A and H_B to isomer **a**, while the signals shifted slightly downfield, to isomer b. The molecules of compounds **IV** and **V** that have less symmetry elements than in the case of compounds **VI**, **VII**, can form more stereoisomers, which apparently results in the complication of their spectra. However, the signals of H_A, H_B, and NH protons in the spectra of these substances are grouped in such a way that a clear analogy is seen with the spectra of diastereomers of compounds **VI** and **VII**.

The assignment of signals for the atoms ^{1}H and ^{13}C in the alkoxylation products **II** and **III** was made using the heteronuclear resonance techniques HMQC (Fig. 1) and HMBC. In the HMBC spectrum of compound **II** the signals of methoxy protons (3.86, 3.78 ppm) of the two diastereomers form two cross-peaks with the carbon atoms signals at 88.87 and 91.82 ppm, respectively. In the HMQC spectrum these carbon signals form cross-peaks with the signals of protons H_B resonating at 4.51 and 4.48 ppm, respectively, confirming the correctness of assigning the latter to H_B and the respective carbon peak, to C^2.



R = CH_3 (**II**), C_2H_5 (**III**); *ortho-* (**IV**), *meta-* (**V**), *para-* (**VI**).

It is evident from the correlation HMQC spectrum of 1-bromo-2-methoxy-1- nitro-1,1,1-trichloropropane **II** that the signal of the H_A proton, which is shifted downfield from the signal of H_B proton in the spectra of both diastereomers, corresponds to the upfield carbon signals. The signal of the carbon atom C^1 at 80.71 corresponds to the proton H_A [$\delta(H_A)$ 6.47 ppm]. In the spectra of the second diastereomer both the proton H_A (6.19 ppm) and the corresponding to it carbon atom C^1 (75.99 ppm) resonate in the stronger field.

In the ^1H NMR spectrum of compound **VII** the signals of the protons H_A, H_B, and NH appear as doublets at 6.87, 5.41, and 4.52 ppm, respectively (diastereomer a), and at 6.75, 4.89, and 5.07 ppm (diastereomer b). The ^{13}C NMR spectrum of this

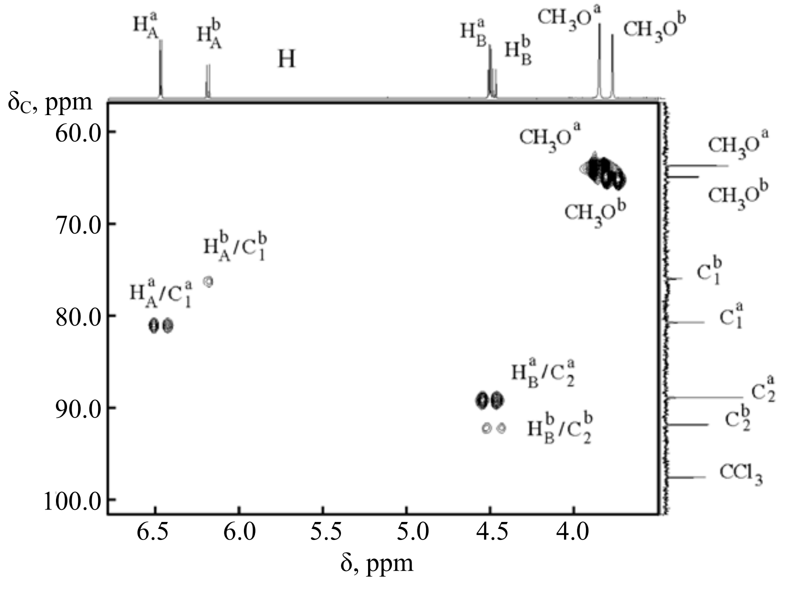


Fig. 1. HMQC spectrum of 1-bromo-2-methoxy-1-nitro-1,1,1-trichloropropane **II** ($CDCl_3$).

compound also contains two set of signals of the carbon atoms: at 83.40 (C^1), 69.68 (C^2), 99.55 ppm (CCl_3) for diastereomer **a**, and at 75.87 (C^1), 73.03 (C^2), 99.08 ppm (CCl_3) for diastereomer b.

In the IR spectra of adducts **II**–**VII** there are absorption bands of symmetrical (1350–1355 cm^{-1}) and asymmetrical (1565–1575 cm^{-1}) vibrations of the nonconjugated nitro group with the separation of the n_s and ν_{as} bands (215–225 cm^{-1}) characteristic of the compounds with geminal nitro group and halogen atom [7, 8]. In the IR spectra of compounds **II** and **III** a strong band of C–O bond is observed at 1115–1125 cm^{-1} characteristic of ethers. In the spectra of the diadducts **IV**–**VII** the absorption bands of aromatic rings (1605–1615 cm^{-1}) and amino group (3375–3420 cm^{-1}) are present.

Recent studies showed that the products of alkylation of aniline and its derivatives with 1-bromo-1-nitro-3,3,3-trichloropropene **I** made possible an approach to aziridines containing nitro- and trichloromethyl groups [3]. It is known that aziridine is a good alkylating agent, and that a number of its derivatives manifest the high mutagenic and anticancer action [9–11].

Aiming to extend the range of application of the previously developed procedure [3], we attempted to carry out the synthesis of aziridines by the example of obtained bisadducts **IV**–**VII**.

The reflux of compounds **V** and **VI** with the solution of potassium acetate in alcohol was found to end by the formation of oily substances, whose purification and identification failed. However, we succeeded to isolate alkoxy-derivative **III** by treating diadduct **VI** with potassium acetate under the same conditions. Its formation is probably connected with the initial deamination of compound **VI** followed by alkoxylation of the formed bromonitroalkene **I**.

$$\mathbf{VI} \xrightarrow[\text{deamination}]{CH_3COOK,\ EtOH,\ \Delta} [\mathbf{I}] \longrightarrow \mathbf{III}$$

The reaction of the diadduct **VII** with potassium acetate gives rise to crude crystalline bis-aziridine **VIII** (71%); after the purification by column chromatography the yield decreased to 32%. Low yield of compound **VIII** can be apparently explained by its partial decomposition on the silica gel that has been earlier registered at the workup of other nitroaziridines [12].

The ^1H and ^{13}C NMR spectra characterize the obtained bis-aziridine **VIII** as diastereomerically uniform compound (Fig. 2). Its vicinal methine protons give rise in the spectrum to unsplit singlets at 4.26 (H_B) and 5.60 ppm (H_A), as in the spectra of the earlier reported 1-aryl-2-nitro-3-trichloromethylaziridines [3] and 1-nitro-2-trichloromethyloxirane [13]. This fact shows the transoid arrangement of the protons in both aziridines, since the value of *cis* spin–spin coupling constant in these heterocycles as a rule exceeds 4 Hz [14]. The correctness of assigning the methine protons signals of aziridine ring is confirmed by the proximity of the obtained data and the corresponding characteristics of the related compounds [3, 15–17]. The signals of the atoms ^1H and ^{13}C in bisaziridine **VIII** were assigned using the heteronuclear resonance HMQC (Fig. 3). Thus, in the HMQC spectrum of this compound there are two cross-peaks corresponding to the correlations of the signal H_B proton (4.26 ppm) with the signal of the carbon atom C^2 (56.42 ppm), and also of the signal of H_A proton (5.60 ppm) with the signal of the carbon atom C^1 (71.93 ppm).

The UV spectrum of bis-aziridine **VIII** contains the intensive absorption band of π→π* transition in the aromatic rings at λ_{max} 269 nm (ε 20400), and also the band of low intensity of π→π* transition at 337 nm (ε 3200).

Thus, 1-bromo-1-nitro-3,3,3-trichloropropene was found to react effectively with the representatives of

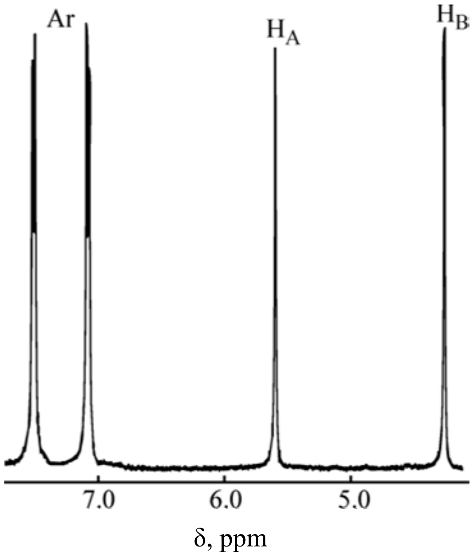


Fig. 2. ^1H NMR spectrum of 4,4'-bis(2-nitro-3-trichloromethylaziridin-1-yl)diphenyl **VIII** ($CDCl_3$).

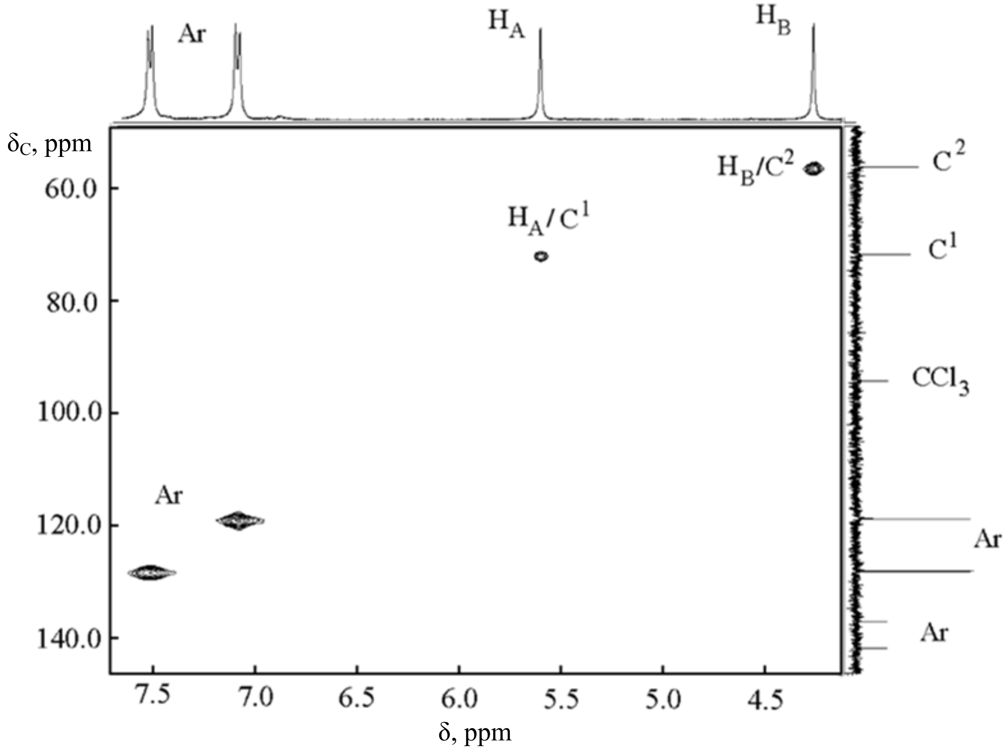


Fig. 3. HMQC spectrum of 4,4'-bis(2-nitro-3-trichloromethylaziridin-1-yl)diphenyl **VIII** ($CDCl_3$).

aliphatic alcohols and aromatic diamines to give the products of nucleophilic addition in high yields. On the basis of the dehydrohalogenation reaction of the product of amination of 1-bromo-1-nitro-3,3,3-trichloropropene with benzidine we realized the synthesis of the first representative of bisaziridines containing nitro- and trichloromethyl groups in the heterocycle. The conclusion was made on the *trans*-orientation of the protons H_A and H_B, and consequently on the *trans*-location of nitro and trichloromethyl groups relative to the plane of aziridine ring.

EXPERIMENTAL

The IR spectra were registered on an InfraLUM FT-02 spectrophotometer from solutions in chloroform (**II–V**, **VII**, **VIII**) and mull with mineral oil (**VI**).

The ^1H and ^{13}C NMR spectra were recorded on a Jeol JNM-ECX400A instrument at operating frequencies 399.78 (^1H) and 100.525 MHz (^{13}C) with chloroform-*d* as a solvent. The signals of the residual protons of the solvent were used as reference.

The UV spectrum was recorded on a Shimadzu UV-2401PC spectrometer in ethanol using quartz cell (l 1 mm, c 8.9×10^{-6} mol l^{-1}). The mass spectra were registered on a MX 1321 mass spectrometer (70 eV, temperature of ionizing chamber 180°C). The elemental analysis was performed on a EuroVector EA 3000 analyzer. The reaction progress was monitored by TLC on Silufol UV-254 plates, detecting with chromatoscope.

The starting 1-bromo-1-nitro-3,3,3-trichloropropene **I** was prepared by the earlier described procedure [2].

1-Bromo-2-methoxy-1-nitro-3,3,3-trichloropropane (II) (a mixture of diastereomers, a:b = 2:1). A solution of 4.81 of 1-bromo-1-nitro-3,3,3-trichloropropene **I** in 20 ml of anhydrous methanol was refluxed for 1.5 h to decolorizing. Then the solvent was removed, and the residue (5.18 g, 96%) was distilled at a reduced pressure. The fraction boiling at 94–97°C (3 mm Hg) was collected. Yield 2.86 g (53%), colorless liquid, n_D^{20} 1.5150 {bp 105°C (1.6 mm Hg) [6]}. IR spectrum, ν, cm^{-1}: 1575, 1355 (NO_2), 1125 (C–O), 810 (CCl_3). ^1H NMR spectrum, δ, ppm: diastereomer **IIa**, 3.86 s (3H, OCH_3), 4.51 d [1H, H_B, $^3J(H_AH_B)$ 4.17 Hz], 6.47 d [1H, H_A, $^3J(H_AH_B)$ 4.17 Hz]; diastereomer **IIb**, 3.78 s (3H, OCH_3), 4.48 d [1H, H_B, $^3J(H_AH_B)$ 6.71 Hz], 6.19 d [1H, H_A, $^3J(H_AH_B)$

6.71 Hz]. ^{13}C–{^{1}H} NMR spectrum, δ_C, ppm: diastereomer **IIa**, 63.71 (OCH_3), 97.56 (CCl_3), 88.87 (C^2), 80.71 (C^1); diastereomer **IIb**, 64.94 (OCH_3), 97.56 (CCl_3), 91.82 (C^2), 75.99 (C^1). Found, %: N 4.54. $C_4H_5BrCl_3NO_3$. Calculated, %: N 4.65.

1-Bromo-1-nitro-3,3,3-trichloro-2-ethoxypropane (III) (a mixture of diastereomers, a:b = 2:1) was similarly prepared from 0.919 g of 1-bromo-1-nitro-3,3,3-trichloropropene **I** and 10 ml of ethanol. Yellowish transparent liquid (1.015 g, 94%) was isolated and subjected to chromatography on silica gel. 1-Bromo-1-nitro-3,3,3-trichloro-2-ethoxypropane (**III**) was eluted with *n*-hexane. Yield 0.616 g (57%), R_f 0.60 (hexane–acetone, 5:1). IR spectrum, ν, cm^{-1}: 1575, 1350 (NO_2), 1115 (C–O), 805 (CCl_3). ^{1}H NMR spectrum, δ, ppm: diastereomer **IIIa**, 1.33 t (3H, CH_3), 4.06 q (2H, OCH_2), 4.59 d [1H, H_B, $^3J(H_AH_B)$ 4.27 Hz], 6.46 d [1H, H_A, $^3J(H_AH_B)$ 4.27 Hz]; diastereomer **IIIb,** 1.20 t (3H, CH_3), 3.85 m, 4.11 m (2H, OCH_2), 4.55 d [1H, H_B, $^3J(H_AH_B)$ 6.41 Hz], 6.19 d [1H, H_A, $^3J(H_AH_B)$ 6.41 Hz]. ^{13}C–{^{1}H} NMR spectrum, δ_C: diastereomer **IIIa**, 15.04 (CH_3), 72.25 (OCH_2), 97.80 (CCl_3), 87.57 (C^2), 81.16 (C^1); diastereomer **IIIb,** 15.17 (CH_3), 73.77 (OCH_2), 97.80 (CCl_3), 90.67 (C^2), 76.40 (C^1). Mass spectrum, *m/z*: 198 [$M^+ - CCl_3$], 152 [$M^+ - CCl_3 - NO_2$].

***N*,*N'*-Bis[1-(bromonitromethyl)-2,2,2-trichloroethyl]benzene-1,2-diamine (IV)**. To a solution of 1.13 g of 1-bromo-1-nitro-3,3,3-trichloropropene **I** in 5 ml of methanol was added a solution of 0.23 g of *o*-phenylene diamine in 10 ml of methanol. This mixture was kept for 3 h, and poured on the crushed ice. The formed yellowish green precipitate suffered tarring when ice melted. The aqueous solution of the oil was extracted with diethyl ether (2 × 25 ml), the extract was dried with $MgSO_4$. The solvent was removed. Yield 1.04 g (76%). The dark red oil obtained was subjected to chromatography on silica gel. The fractions eluted with *n-h*exane and carbon tetrachloride afforded 0.86 g (63%) of dark red oily substance **IV** (a mixture of diastereomers), R_f0.48 (hexane–acetone, 2:1). IR spectrum, ν, cm^{-1}: 3375 (NH), 1605, 1520 (Ar), 1575, 1350 (NO_2), 810 (CCl_3). ^{1}H NMR spectrum, δ, ppm: diastereomer **IVa**, 4.43–4.78 m (1H, NH), 5.30–5.39 m (1H, H_B), 6.91–7.00 m (1H, H_A), 6.91–7.00 m (4H, Ar); diastereomer **IVb**, 4.93–5.15 m (1H, NH), 4.83–4.93 m (1H, H_B), 6.81–6.88 m (1H, H_A), 6.91–7.00 m, 7.05–7.10 m (4H, Ar). Mass spectrum: *m/z* 646 [M^+].

***N*,*N'*-Bis[1-(bromonitromethyl)-2,2,2-trichloroethyl]benzene-1,3-diamine (V)** was prepared similarly from 1.17 g of 1-bromo-1-nitro-3,3,3-trichloropropene **I** and 0.23 g of *m*-phenylenediamine in 15 ml of methanol. The yellowish green precipitate was filtered off. Yield 1.31 g (94%) (a mixture of diastereomers), mp 44–46°C. IR spectrum, ν, cm^{-1}: 3420 (NH), 1615, 1520 (Ar), 1575, 1350 (NO_2). ^{1}H NMR spectrum, δ, ppm: diastereomer **Va**, 4.44–4.50 m (1H, NH), 5.34–5.39 m (1H, H_B), 6.84–6.87 m (1H, H_A), 6.26–6.42 m, 7.06–7.16 m (4H, Ar); diastereomer **IVb**, 5.02–5.05 m (1H, NH), 4.79–4.86 m (1H, H_B), 6.74–6.75 m (1H, H_A), 6.26–6.42 m, 7.06–7.16 m (4H, Ar). Mass spectrum: *m/z* 646 [M^+]. Found, %: C 22.62; H 2.00; N 8.45. $C_{12}H_{10}Br_2Cl_6N_4O_4$. Calculated, %: C 22.28; H 1.56; N 8.66.

***N*,*N'*-Bis[1-(bromonitromethyl)-2,2,2-trichloroethyl]benzene-1,4-diamine (VI)** was prepared similarly from 1.01 g of 1-bromo-1-nitro-3,3,3-trichloropropene **I**, 0.2 g of *p*-phenylenediamine in 15 ml of methanol. The reaction mixture was kept for 1 h. The yellow precipitate was filtered off and washed with methanol. Yield 0.68 g (56%) (diastereomers mixture, a:b = 3:1), mp 174–175°C (decomp.). After 3 days additional 0.12 g (9%) of compound **VI** was isolated from the mother liquor. Overall yield 65%. IR spectrum, ν, cm^{-1}: 3390 (NH), 1520 (Ar), 1565, 1350 (NO_2). ^{1}H NMR spectrum, δ, ppm: diastereomer **VIa**, 4.25 d [1H, NH, $^3J(NHH_B)$ 10.99], 5.23 d.d [1H, H_B, ^3J (H_AH_B) 2.44, $^3J(NHH_B)$ 10.99 Hz], 6.85 d [1H, H_A, ^3J (H_AH_B) 2.44 Hz], 6.71–6.81 m (4H, Ar); diastereomer **VIb**, 4.74 d [1H, NH, $^3J(NHH_B)$ 3.36 Hz], 4.74 d.d [1H, H_B, $^3J(H_AH_B)$ 2.75, $^3J(NHH_B)$ 3.36 Hz], 6.69 d [1H, H_A, $^3J(H_AH_B)$ 2.75 Hz], 6.73–6.81 m (4H, Ar). Mass spectrum: *m/z* 646 [M^+]. Found, %: C 21.83; H 1.99; N 8.18. $C_{12}H_{10}Br_2Cl_6N_4O_4$. Calculated, %: C 22.28; H 1.56; N 8.66.

***N*,*N'*-Bis[1-(bromonitromethyl)-2,2,2-trichloroethyl]diphenyl-4,4'-diamine (VII)** was prepared similarly from 0.95 g of 1-bromo-1-nitro-3,3,3-trichloropropene **I**, 0.32 g benzidine in 15 ml of methanol. The reaction mixture was kept for 1.5 h and poured on the crushed ice. The yellowish orange precipitate was filtered off. Yield 1.12 g (88%) (diastereomers mixture, a:b = 2:1), mp 62–65°C. IR spectrum, ν, cm^{-1}: 3420 (NH), 1615, 1510 (Ar), 1575, 1350 (NO_2), 815 (CCl_3). ^{1}H NMR spectrum, δ, ppm: diastereomer **VIIa**, 4.52 d [1H, NH, $^3J(NHH_B)$ 10.99 Hz], 5.41 d.d [1H, H_B, $^3J(H_AH_B)$ 2.14, $^3J(NHH_B)$ 10.99 Hz], 6.87 d [1H, H_A, $^3J(H_AH_B)$ 2.14 Hz], 6.85–6.92 m, 7.39–7.46 m (4H, Ar); diastereomer **VIIb**, 5.07 d [1H, NH, $^3J(NHH_B)$ 10.68 Hz], 4.89 d.d [1H, H_B, $^3J(H_AH_B)$

3.05, ^3J(NHH$_B$) 10.68 Hz], 6.75 d [1H, H$_A$, ^3J(H$_A$H$_B$) 3.66 Hz], 6.85–6.92 m, 7.39–7.46 m (4H, Ar). ^{13}C–{^{1}H} NMR spectrum, δ_C, ppm: diastereomer **VIIa**, 69.68 (C^2), 83.40 (C^1), 99.55 (CCl$_3$), 114.81, 127.75, 133.28, 143.60 ($C_{12}H_8$); diastereomer **VIIb**, 73.03 (C^2), 75.87 (C^1), 99.08 (CCl$_3$), 114.53, 127.75, 133.09, 144.56 ($C_{12}H_8$). Mass spectrum: *m/z* 722 [M^+]. Found, %: C 30.45; H 2.46; N 7.58. $C_{18}H_{14}Br_2Cl_6N_4O_4$. Calculated, %: C 29.91; H 1.95; N 7.75.

4,4'-Bis(2-nitro-3-trichloromethylaziridin-1-yl)-biphenyl (VIII). To a boiling solution of 1.12 g of diadduct **VII** in 15 ml of ethanol was added a hot solution of 0.23 g of potassium acetate in 15 ml of ethanol. The reaction mixture was refluxed for 4 h and poured on the crushed ice. The reddish substance (0.62 g, 71%) was filtered off and subjected to chromatography on silica gel eluting with *n*-hexane to give 0.27 g (32%) of greenish crystals, mp 154–156°C (petroleum ether). IR spectrum, ν, cm^{-1}: 1610, 1495 (Ar), 1560, 1350 (NO$_2$), 815 (CCl$_3$). ^{1}H NMR spectrum, δ, ppm: 4.26 s (1H, H$_B$), 5.60 s (1H, H$_A$), 7.08 d, 7.51 d (8H, $C_{12}H_8$). ^{13}C–{^{1}H} NMR spectrum, δ_C, ppm: 56.42 (C^2), 71.93 (C^1), 94.42 (CCl$_3$), 118.97, 128.27, 137.20, 141.99 ($C_{12}H_8$). UV spectrum, λ_{max}, nm (ε): 269 (20400), 337 (3200). Mass spectrum: *m/z* 560 [M^+]. Found, %: C 38.58; H 2.98. $C_{18}H_{12}Cl_6N_4O_4$. Calculated, %: C 38.53; H 2.16.

Reaction of diadduct VI with potassium acetate. To a boiling solution of 1.3 g of compound **VI** in 15 ml of ethanol was added a hot solution of 0.3 g of potassium acetate in 15 ml of ethanol. The reaction mixture was refluxed for 4.5 h and poured into the crushed ice. The emulsion formed was extracted with chloroform, dried over magnesium sulfate, and evaporated. The formed dark red viscous substance (0.74 g) was subjected to chromatography on silica gel eluting with *n*-hexane to give 0.25 g (38%) of colorless oil **III** as a diastereomers mixture (a:b = 2:1), R_f 0.60 (hexane–acetone, 5:1). The IR and ^{1}H NMR spectral data are corresponded to that for 1-bromo-1-nitro-3,3,3-trichloro-2-ethoxypropane **III**.

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