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Reaction of *N*-Phenyltriflamide with 1,2-Dibromoethane and Propargyl Bromide. Unexpected Cleavage of C–C and C–N Bonds

B. A. Shainyan* and Yu. S. Danilevich

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

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Abstract—Reaction of *N*-phenyltriflamide with 1,2-dibromoethane under basic conditions in DMSO unexpectedly results in *N*-methyl-*N*-phenyltriflamide and 1,3-diphenylurea. The presumed reaction mechanism includes the formation of unstable intermediate disubstitution product TfN(Ph)CH_2CH_2N(Ph)Tf that suffers the the C–C bond cleavage resulting in TfN(Me)Ph and *N*,*N*′-methanediylbis(*N*-phenyltriflamide). The latter reacts with K_2CO_3 releasing two molecules of potassium triflinate and after hydrolysis of diphenylcarbodiimide PhN=C=NPh gives 1,3-diphenylurea. With propargyl bromide, N-phenyltriflamide affords *N*-propargyl-*N*-phenyltriflamide in high yield. The bromination of the latter results in a mixture of *Z*,*E*-isomers of *N*-(2,3-dibromoprop-2-en-1-yl)-*N*-phenyltriflamide which undergo dehydrobromination giving first *N*-(3-bromopropanedienyl)-*N*-phenyltriflamide and then the products of the C–N bond cleavage: *N*-phenyltriflamide and 3,3-dimethoxyprop-1-yne.

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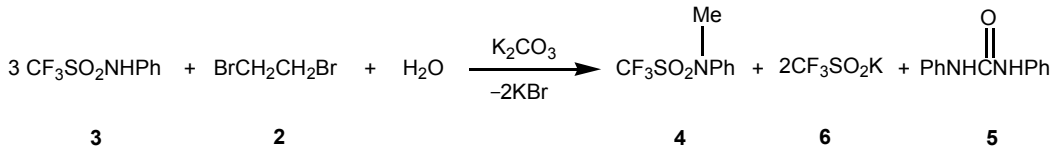
Unsaturated derivatives of triflamide of general formula TfNHR (Tf = CF_3SO_2) with substitutent R containing one or more multiple bonds are interesting both from the point of view of their synthetic potential, and of possible tautomerism in these compounds. Until recently, the family of triflamide derivatives and other perfluoralkanesulfonamides was small and consisted mostly of imines of the type $R_FSO_2N{=}CHAr$ [1–5]. After unsuccessful attempt to obtain first vinyl triflamide derivative by dehydro-bromination of TfNHCH$_2$CHBrPh that resulted in a substituted piperazine [6], we were able to synthesize (*E*)-TfN(Me)CH=CHPh from its methyl analog [7]. In subsequent studies we synthesized a series of *N*-vinyl [8, 9], *N*-allyl [10, 11], *N*-allenyl [12], *N*-propargyl [13], and other highly unsaturated derivatives of triflamide [14–17].

As for disubstituted linear unsaturated derivatives of triflamide, except for the synthesis of *N*,*N*′-hexa-2,4-diyne-1,6-diylbis(triflamide) TfNHCH$_2$C≡C–C≡CCH$_2$NHTf [14], we know only one study, in which the isomers of *N*,*N*′-but-2-en-1,4-diylbis(triflamide) TfNHCH$_2$CH=CHCH$_2$NHTf have been described [18].

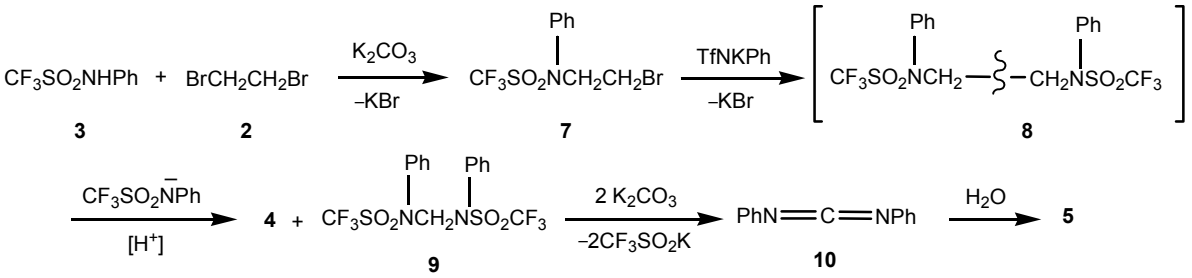
Aiming to extend the series of unsaturated derivatives of triflamide we investigated in the present study the reaction of N-phenyltriflamide with 1,2-dibromoethane expecting to synthesize *N*,*N*′-ethane-1,2-diylbis(*N*-phenyltriflamide) TfN(Ph)CH_2CH_2N(Ph)Tf and to transform it into unsaturated derivatives TfN(Ph)CH=CHN(Ph)Tf and/or TfN(Ph)C≡CN(Ph)Tf by subsequent bromination–dehydrobromination. We also investigated the reaction of *N*-phenyltriflamide with propargyl bromide and explored the bromination–dehydrobromination of the formed *N*-propargyl-*N*-phenyltriflamide TfN(Ph)CH$_2$C≡CH.

First we carried out the reaction of triflamide **1** with 1,2-dibromoethane **2** in DMSO in the presence of equimolar amount of $KHCO_3$. However only intractable mixture of compounds was obtained, in its ^{19}F NMR spectrum up to eight signals were present. We assumed that the reason of complicated course of the reaction was the presence of a free NH proton in the forming salt CF_3SO_2NHK, so we replaced triflamide with its *N*-phenyl-substituted analog **3** obtained by the reaction of triflic anhydride with aniline, using the latter both as the reactant and the solvent. The reaction of *N*-

Scheme 1.



Scheme 2.



phenyltriflamide **3** with 1,2-dibromo-ethane **2** in DMSO in the presence of K_2CO_3 proceeds smoothly but with completely unexpected result. Two products of the reaction were isolated: *N*-methyl-*N*-phenyltriflamide **4** and 1,3-diphenylurea **5**. As shown in Scheme 1, the reaction may be equated assuming that two molecules of *N*-phenyltriflamide potassium salt react with one molecule of 1,2-dibromoethane, and the third molecule is reduced to salt **6**.

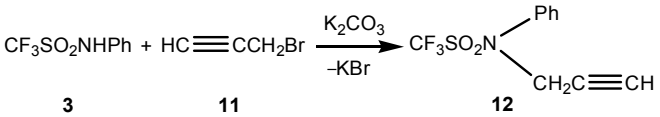
^{1}H NMR spectrum of the reaction mixture before separation contains two triplet signals at 3.5 and 4.3 ppm with a spin-spin coupling constant of 6.6 Hz, evidencing the formation of monosubstituted compound **7** (Scheme 2). Its further reaction with potassium salt of amide **3** gives the product of disubstitution **8** that decomposes under the action of the third molecule of salt TfNKPh as shows the presence of a methyl group in the spectrum of the reaction product **4**. Further elimination of two molecules of trifluormethanesulfinic acid CF_3SO_2H from compound **9** in the form of potassium triflinate results in diphenylcarbodiimide **10** that hydrolyses giving the final product, 1,3-diphenylurea **5**. Due to the presence of two triflamide residues in molecule **9** the CH_2 group becomes more acidic which favors the elimination. The triflinate anion may be oxidized to triflate under the action of the solvent (DMSO). The formation of 1,3-diphenylurea **5** is confirmed by the identity of its IR, ^{1}H, and ^{13}C NMR spectra with the spectra of an authentic sample.

We tried to perform the reaction of *N*-phenyltriflamide **3** with 1,2-dichloroethane in similar conditions, however the reaction did not occur even at 10-hour heating at 80°C: only the initial substrate **3** was recovered. Obviously it is the result of the lower nucleofugality of the chlorine atom compared to bromine.

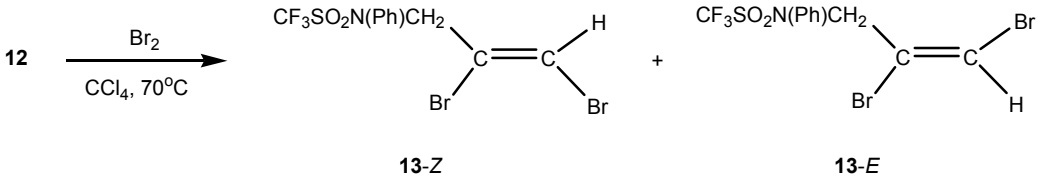
Unlike the reaction with 1,2-dibromoethane **2**, *N*-phenyltriflamide **3** is alkylated easily and in high yields with propargyl bromide **11** in the presence of potassium carbonate at room temperature providing a new highly unsaturated derivative of triflamide, *N*-propargyl-*N*-phenyltriflamide **12** (Scheme 3).

The bromination of alkyne **12** gives a mixture of *Z*- and *E*-isomers of *N*-(2,3-dibromoprop-2-en-1-yl)-*N*-

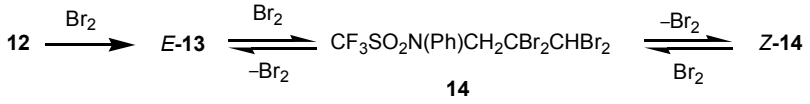
Scheme 3.



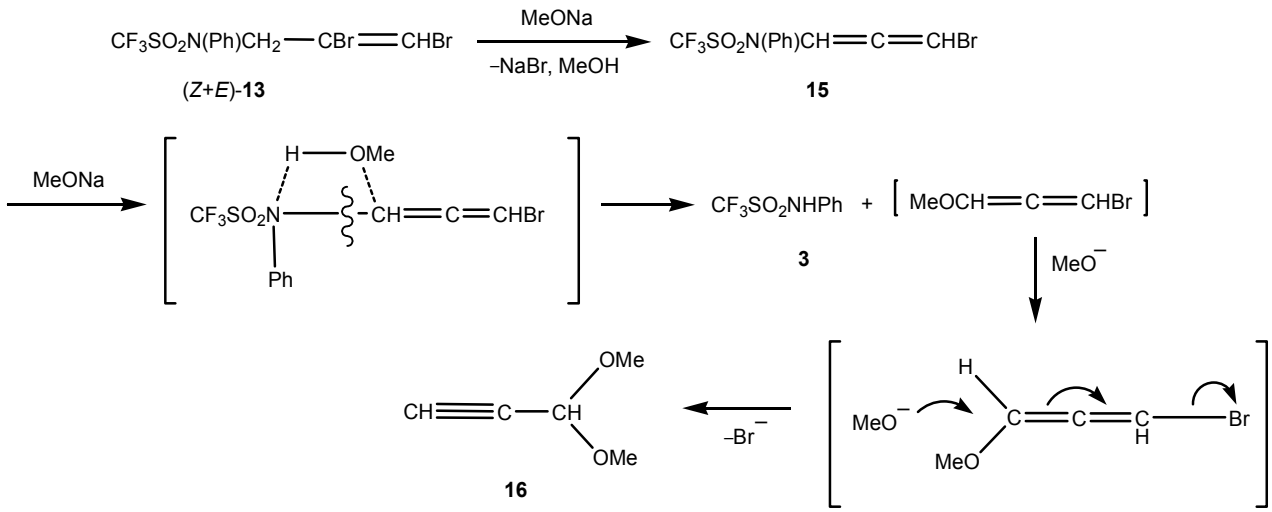
Scheme 4.



Scheme 5.



Scheme 6.



phenyltriflamide TfN(Ph)CH_2CBr=CHBr **13**. No diadduct is formed (Scheme 4).

We previously demonstrated that the bromination of *N*-propargyltriflamide gave a mixture of *Z*- and *E*-isomers of monoadduct in a ratio 24 : 76; the product of dibromoination did not form even at excess of bromine [16]. The formation of two isomers was proved by the presence of two sets of signals from the fragment CH_2CBr=CHBr in ^{13}C NMR spectrum and two close lying signals of CF_3 groups in ^{13}C and ^{19}F spectra. The assignment of the signals to the *Z*- and *E*-isomers was performed by measuring the coupling constants $^3J_{C–H}$ in the allyl fragment C–C=C–H (3.1 Hz in *Z*- and 6.9 Hz in *E*-isomer).

Interesting results were obtained by monitoring the isomeric composition at varying the reaction conditions. With the deficiency of bromine, when conversion of compound **12** did not exceed 30%, the ratio *Z*-**13**/*E*-**13** measured with ^{1}H NMR method was 7 : 93. Adding bromine and increasing the conversion to 85% this ratio increases to 21 : 79, and the ratio attains 35 : 65 at the full conversion. Further heating with bromine excess changes this ratio in favor of *Z*-isomer till *Z*-**13**/*E*-**13** = 55 : 45 that then remains constant, showing the reached equalibrium. Hence it may be concluded that isomers *Z*-**13** and *E*-**13** are formed in different ways: the *E*-isomer is formed by bromination of compound **12**, while the *Z*-isomer is, probably, formed by debromination of the intermediate unstable adduct **14** (Scheme 5).

Consequently, *E*-isomer is a kinetically controlled reaction product, while *Z*-isomer is more stable thermodynamically. The initial formation of *E*-isomer is consistent with the rule of *trans*-addition, whereas the higher stability of *Z*-isomer is not so obvious. Quantum-chemical calculations by DFT/B3LYP/6-311G(d,p) and MP2/6-311G(d,p) methods showed the energy preference of *Z*-**13**-isomer compared to *E*-**13**-isomer by 1.03 (DFT) and 1.17 kcal mol^{-1} (MP2). The good consistency of the calculation results both with each other, and with experimentally observed prevalence of *Z*-isomer confirms the suggested mechanism. The calculations also confirm the instability of diadduct **14**: although a local minimum on the potential energy surface corresponds to molecule **14**, its formation from *Z*-isomer and bromine molecule is endothermic by 11.8 kcal mol^{-1}, while the formation of *Z*-isomer itself from compound **12** and bromine molecule is exothermic by 34.8 kcal mol^{-1}.

A higher stability of the *Z*-**13** versus *E*-**13** isomer is, apparently, due to strong electron acceptor character of the CF_3SO_2 group, since the MP2/6-311G calculated for comparison energy difference between the *Z*- and *E*-isomers of *N*-(2,3-dibromoprop-2-en-1-yl)-*N*-methylaniline MeN(Ph)CH_2CBr=CHBr is as low as

0.13 kcal mol^{-1}, which is much smaller than the above value of 1.17 kcal mol^{-1} for the isomers of compound **13**.

The dehydrobromination of monoadducts *Z*-**13** and *E*-**13** may result in either allenyl product TfN(Ph)·CH=C=CHBr, or propargyl product TfN(Ph)CH$_2$·C≡CBr that are easy to distinguish by ^{1}H and ^{13}C NMR spectra, or to its mixture (Scheme 6). Actually, at performing the reaction with a deficiency of sodium methyloxide (about 1/3 of stoichiometric amount), allene **15** was detected by the presence of two doublets in ^{1}H NMR spectrum at 7.06 (=CHBr) and 6.25 ppm (NCH=), ^4J 5.1 Hz. However at a small excess of sodium methyloxide instead of elimination product of allenyl or propargyl type unexpectedly products were obtained of the rupture of the N–C bond: *N*-phenyltriflamide **3** and 3,3-dimethoxyprop-1-yne **16**. These results may be explained in the framework of a reaction mechanism including an intermediate formation of 3-bromopropadienyl derivative **15** and its subsequent fragmentation and bromine substitution with the transition of the reaction center. The attack of the methoxide ion in the last stage on the position *3* of intermediate 1-bromo-3-methoxypropadiene is assisted by the conjugation of the double C=C bond with methoxy substituent that polarizes it in the direction from the oxygen atom to the bromine atom. The last stage as evident from Scheme 6 is similar to the well-known mechanism of nucleophilic substitution S_N2'.

The formation of *N*-phenyltriflamide is confirmed by practically full coincidence of ^{13}C NMR spectrum of reaction product formed by Scheme 6 with the spectrum of authentic sample **3**. The structure of 3,3-dimethoxyprop-1-yne **16** is proved by ^{1}H, ^{13}C, and ^{13}C{^{1}H} NMR spectra. The proton spectrum of compound **16** contains a singlet of two methoxy groups at 3.42 ppm (6H) and two doublets of one-proton intensity each at 2.57 and 5.19 ppm (*J* 1.8 Hz), fully consistent with the published data [19]. ^{13}C{^{1}H} NMR spectrum contains signals of methyl groups (d.d, 53.54 ppm, *J* 143.5, 5.1 Hz), of the terminal acetylene carbon atom (d, 74.50 ppm, *J* 252.8 Hz), another acetylene carbon atom (s, 77.60 ppm), and of acetal methine carbon atom (d.q, 92.67 ppm, *J* 169.6, 4.5 Hz) also in good agreement with the published data [19] that unambiguously proves the assumed structure and the reaction mechanism.

Considering the results of our previous investigations it may be concluded that the direction of dehydrobromination of *N*-substituted *N*-(2-bromoallyl)-triflamides TfN(R)CH$_2$CBr=CHR' is quite sensitive even to small structural variations. So, the reaction of *N*-benzyl-*N*-(2-bromoallyl)triflamide TfN(Bn)CH$_2$CBr=CH$_2$ with 10-fold excess of sodium methylate ends at the stage of formation of the corresponding allene TfN(Bn)·CH=C=CH$_2$ [11]. Unlike this, the reaction of compound **13** which with the deficiency of sodium methylate gives similar allenyl product, in the presence of only 25% excess of the base goes further resulting in the products of the N–C bond rupture **3** and **16**. Evidently, the effect of electronegative groups (Ph comparing to Bn, and Br comparing to H) is small, but enough to provide the bond rupture, especially in combination with the subsequent substitution of the bromine atom.

Hence, an unusual direction of reaction of *N*-phenyltriflamide with 1,2-dibromoethane resulting in the rupture of C–C bond in the latter and in the formation of *N*-methyl-*N*-phenyltriflamide and 1,3-diphenylurea was discovered. A reaction mechanism was suggested including the formation of unstable disubstitution product TfN(Ph)CH$_2$CH$_2$N(Ph)Tf, its decomposition under the action of salt molecule TfNKPh, release of two molecules of trifluormethanesulfine acid as potassium salt from the formed *N*,*N*'-methanediylbis(*N*-phenyltriflamide), and the final stage of hydrolysis of diphenylcarbodiimide. The reaction of *N*-phenyltriflamide with propargyl bromide gives new unsaturated derivative of triflamide, *N*-propargyl-*N*-phenyltriflamide that undergoes bromination giving a mixture of isomeric monoadducts which are kinetically and thermodynamically controlled products formed in different ways.

EXPERIMENTAL

IR spectra were recorded on a spectrophotometer Bruker Vertex 70. ^{1}H, ^{13}C, and ^{19}F NMR spectra were registered on a spectrometer Bruker DPX 400 at working frequencies 400 (^{1}H), 100 (^{13}C), and 376 (^{19}F) MHz in $CDCl_3$ (if not indicated otherwise). Chemical shifts in ^{1}H and ^{13}C NMR spectra are reported with respect to TMS, in ^{19}F NMR spectra, with respect to with $CFCl_3$. Solvents were dried and purified before use by standard methods.

Quantum-chemical calculations were performed applying the software package GAUSSIAN09 [20] by B3LYP or MP2 methods using the basis set 6-311G(d,p) with full optimization of geometry.

Reaction of triflamide with 1,2-dibromoethane. To a solution of 0.5 g of triflamide in 10 mL of water was added while stirring a solution of 0.3 g of K_2CO_3 in 10 mL of water, the mixture was stirred for 30 min, evaporated at a reduced pressure, the residue was dried in a vacuum. To the obtained salt TfNHK was added 20 mL of DMSO and 0.31 g (0.14 mL) of 1,2-dibromoethane, the reaction mixture was stirred for 6 h, washed with water to remove DMSO, extracted with chloroform. The extract was dried with $MgSO_4$, evaporated in a vacuum, the residue was analyzed by the NMR method. In ^{1}H NMR spectrum several signals of NH groups are present in the region of 7.8–8.7 ppm and a broad signal at 3.3–3.9 ppm, ^{13}C NMR spectrum contains 5–6 signals in the region of 40–54 ppm and few quartets of CF_3 groups at 120 ppm, and ^{19}F NMR spectrum contains 7–8 signals in the region of –78 to –73 ppm. It was impossible to isolate individual compounds by column chromatography.

Reaction of *N*-phenyltriflamide with 1,2-dibromoethane. To 15 mL of aniline while stirring was added dropwise 4 g (2,4 mL) of trifluoromethanesulfonic anhydride, the mixture was stirred for 30 min, acidified with 10% HCl, the precipitate was filtered off, washed with water, dried and sublimated in a vacuum to get 2.9 g (90%) of *N*-phenyltriflamide **3** as a white powder (δ_F –75.30 ppm).

Compound **3** was dissolved in 20 mL of DMSO, 1.2 g (0.55 mL) of 1,2-dibromoethane and 4 g of freshly calcined K_2CO_3 was added, the reaction mixture was stirred for 6 h, then poured in water. The obtained compound after filtering and drying was identified as **1,3-diphenylurea (5)**, mp 240°C. IR spectrum (KBr), ν, cm^{-1}: 3327, 3287 (NH), 3035–3194 (CH), 1648, 1596, 1555 (arom.), 1497, 1445, 1314, 1298, 1233, 894, 755, 699. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 6.97 t (1H, H^p, *J* 7.0 Hz), 7.28 d (2H, H^m, *J* 7.4 Hz), 7.46 d (2H, H^o, *J* 7.7 Hz), 8.65 s (1H, NH). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 118.40 (C^o), 121.97 (C^p), 128.82 (C^m), 139.71 (C^i), 152.69 (C=O). All spectra are identical to the spectra of an authentic sample.

The mother liquor was extracted with chloroform, the extract was washed with water to remove the remnants of DMSO, dried with $MgSO_4$, evaporated, the residue was distilled to give ***N*-methyl-*N*-phenyltriflamide (4)**. Yield 0.5 g (50%), boiling point 63°C (0.8 mm Hg). IR spectrum (thin layer), ν, cm^{-1}: 3100–2740 (CH), 1599 (arom), 1496, 1436, 1379, 1210, 1143, 1009, 951, 752, 695. ^{1}H NMR spectrum, δ, ppm: 2.67 s (3H, CH_3), 7.21–7.28 m (3H, H^{m+p}), 7.29–7.35 m (2H, H^o). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 39.87 (CH_3), 119.92 q (CF_3, *J* 323.1 Hz), 123.10 (C^m), 126.76 (C^p), 129.35 (C^o), 134.58 (C^i). ^{19}F NMR spectrum: δ –75.32 ppm. Found, %: C 40.43; H 3.20; F 23.65; N 5.70; S 13.09. $C_8H_8F_3NO_2S$. Calculated, %: C 40.17; H 3.37; F 23.83; N 5.86; S 13.40.

***N*-Propargyl-*N*-phenyltriflamide (11).** To 20 mL of DMSO was added 3.73 g of *N*-phenyltriflamide and 1.97 g (1.5 mL) of propargyl bromide. After the dissolution of *N*-phenyltriflamide 4 g of K_2CO_3 was added, the formed suspension was stirred for 6 h at room temperature, poured in water, extracted with chloroform, the extract was washed with water, dried with $MgSO_4$, evaporated, the residue was distilled in a vacuum. Yield 3.86 g (88%), boiling point 74°C (0.6 mm Hg). IR spectrum (thin layer), ν, cm^{-1}: 3298 (≡C–H), 3069, 3045, 2985, 2931 (C–H), 2131 (C≡C), 1593 (arom), 1492, 1396, 1311, 1196, 1146, 1076, 1032, 881, 770. ^{1}H NMR spectrum, δ, ppm: 2.41 t (1H, ≡CH, *J* 2.2 Hz), 4.54 d (2H, CH_2, *J* 2.2 Hz), 7.46 s (5H, Ph). ^{13}C NMR spectrum, δ, ppm: 43.83 (NCH_2), 75.45 (CH≡), 77.02 (–C≡), 120.53 q (CF_3, *J* 322.4 Hz), 129.67 (C^o), 130.09 (C^m), 130.22 (C^p), 137.32 (C^i). ^{19}F NMR spectrum: δ –74.06 ppm. Found, %: C 45.43; H 2.97; F 22.02; N 5.60; S 11.81. $C_{10}H_8F_3NO_2S$. Calculated, %: C 45.63; H 3.06; F 21.65; N 5.32; S 12.18.

***N*-(2,3-dibromoprop-2-en-1-yl)-*N*-phenyltrifluoromethanesulfonamide (13).** To 0.5 g (1.9 mmol) of reagent **11** in 20 mL of anhydrous CCl_4 was added 0.31 g (0.19 mmol) of bromine, and the mixture was stirred for 8 h at 70°C, then more 0.05 mL of bromine was added and the stirring while heating was continued for 6 h more. The solvent was removed in a vacuum. The residue contained the mixture of isomers *Z*,*E*-**13**. Found, %: C 27.83; H 1.75; (Br+S) 45.46; F 14.08; N 3.17. $C_{10}H_8Br_2F_3NO_2S$. Calculated, %: C 28.39; H 1.91; Br 37.78; F 13.47; N 3.31; S 7.58.

***Z*-13-Isomer.** ^{1}H NMR spectrum, δ, ppm: 4.67 s (2H, CH_2), 6.73 s (1H, =CHBr), 7.40–7.45 m (5H, Ph). ^{13}C NMR spectrum, δ, ppm: 60.35 t.d (NCH_2, *J* 145.9, 3.1 Hz), 113.87 d.t (=CHBr, *J* 194.1, 5.4 Hz), 118.77 d (CBr=, *J* 22.1 Hz), 120.27 q (CF_3, J_{CF} 324.0 Hz), 129.10 d (C^m, *J* 162.9 Hz), 129.49 d (C^p, *J* 165.1 Hz), 129.83 d (C^o, *J* 162.9 Hz), 135.92 (C^i). ^{19}F NMR spectrum: δ –73.25 ppm.

***E*-13-Isomer.** ^{1}H NMR spectrum, δ, ppm: 4.87 s (2H, CH_2), 6.56 s (1H, =CHBr), 7.40–7.45 m (5H, Ph). ^{13}C NMR spectrum, δ, ppm: 55.85 t.d (NCH_2, *J* 145.5, 6.9 Hz), 109.31 d.t (=CHBr, *J* 202.6, 3.9 Hz), 120.33 q (CF_3, J_{CF} 323.8 Hz), 125.81 d (CBr=, *J* 13.3 Hz), 129.05 d (C^m, *J* 160.0 Hz), 129.44 d (C^p, *J* 163.3 Hz), 129.75 d (C^o, *J* 162.9 Hz), 135.48 (C^i). ^{19}F NMR spectrum: δ –73.20 ppm.

Dehydrobromination of the mixture of *Z*- and *E*-isomers (13). *a*. *With a deficit of sodium methylate*. To solution of 0.3 g (0.7 mmol) of compound **13** in 10 mL of methanol was added 10 mg (0.18 mmol) of sodium methylate, the mixture was stirred for 4 h at 40°C, filtered, the solvent was removed, the residue was analyzed by NMR method.

b. *With excess of sodium methylate*. To a solution of 2.5 g (5.9 mmol) of compound **13** in 30 mL of methanol was added 0.4 g (7.4 mmol) of sodium methylate, the mixture was stirred for 4 h at 40°C, filtered, the solvent was evaporated, the residue was dissolved in water and extracted with chloroform. The extract was dried with $MgSO_4$, the solvent was removed, the residue was analyzed by NMR method.

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