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# Reaction of γ-Dicarboxylic Acids Amides and Imides with Trifluoromethanesulfonamide and Formaldehyde

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**Abstract**—Three-component condensation of trifluoromethanesulfonamide with paraformaldehyde and succinamide depending on the reaction conditions led alongside bis(trifluoromethanesulfonamido)methane to the formation of a substitution product, bis[(trifluoromethylsulfonyl)aminomethyl]succinamide, or to a cyclization product, *N*-[trifluoromethylsulfonyl)aminomethyl]succinimide. The attempt to obtain the latter by the reaction of the trifluoromethanesulfonamide sodium salt $CF_3SO_2NHNa$ with *N*-chloromethylsuccinimide unexpectedly resulted in *N,N*-bis(succinimidomethyl)-trifluoromethanesulfonamide. Analogously the reaction of $CF_3SO_2NHNa$ with *N*-chloromethyl-phthalimide gave *N,N*-bis(phthalimidomethyl)trifluoromethanesulfonamide. The reaction of $CF_3SO_2NHNa$ with succinimide and phthalimide in water and alcohol solution resulted in the ring opening and further transformation of the formed monosubstituted *N*-(trifluoromethylsulfonyl)amides of succinic and phthalic acids.

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We showed formerly that the condensation of trifluoromethanesulfonamide (triflamide) $CF_3SO_2NH_2$ with paraformaldehyde depending on the reaction conditions led to the formation of versatile linear and cyclic products [1, 2]. Also the three-component reactions of triflamide with paraformaldehyde and various amides were investigated, and in some events mixed condensation products were obtained [3]. It was shown in particular that the reaction of triflamide, paraformaldehyde, and malonamide provided a spirocyclic condensation product involving both amido groups and the methylene group of the malonamide, 4,10-bis-(trifluoromethylsulfonyl)-2,4,8,10-tetraazaspiro-[5,5]undecane-1,7-dione [3]. In extension of this research the present study consists in the investigation of the reaction of succinamide, succinimide, and phthalimide with trifluoromethanesulfonamide and formaldehyde under various conditions.

The investigation of the transformations in the system trifluoromethanesulfonamide–formaldehyde–succinamide (**I**) revealed the fundamental dependence of the reaction direction on its conditions. In the medium of concn. $H_2SO_4$ alongside the unreacted triflamide and the linear product of its condensation with formaldehyde [bis(trifluoromethanesulfonamido)methane (**II**)] the formation was found of a product of the triple condensation at both amido groups, bis[(trifluoromethylsulfonyl)aminomethyl]-succinamide (**III**) that was isolated after washing the reaction product **II** with a mixture ether–hexane and was recrystallized from dichloromethane.

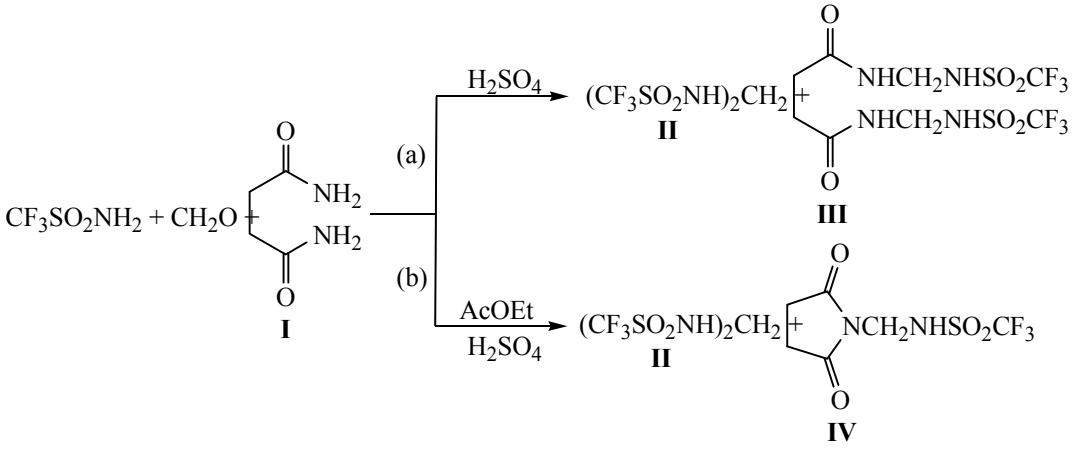
The same reaction carried out in ethyl acetate in the presence of sulfuric acid alongside compound **II** unexpectedly furnished a cyclic amidomethylation product, *N*-[(trifluoromethylsulfonyl)aminomethyl]succinimide (**IV**) isolated analogously and recrystallized from chloroform.

The structure of compound **III** was proved by the presence in its $^1$H NMR spectrum of a singlet from $CH_2CH_2$ group at 2.36 ppm, a broadened singlet of $NHSO_2$, a triplrt of NHCO, and a doublet of $NCH_2N$ in the ratio 2:1:1:2, and of the corresponding signals in the $^{13}$C NMR spectrum. Unlike that in the $^1$H NMR spectrum of compound **IV** appeared a singlet of $CH_2CH_2$ group at 2.68 ppm, and signals of $NHSO_2$ and $NCH_2N$ groups in the ratio 4:1:2, and the corresponding signals in the

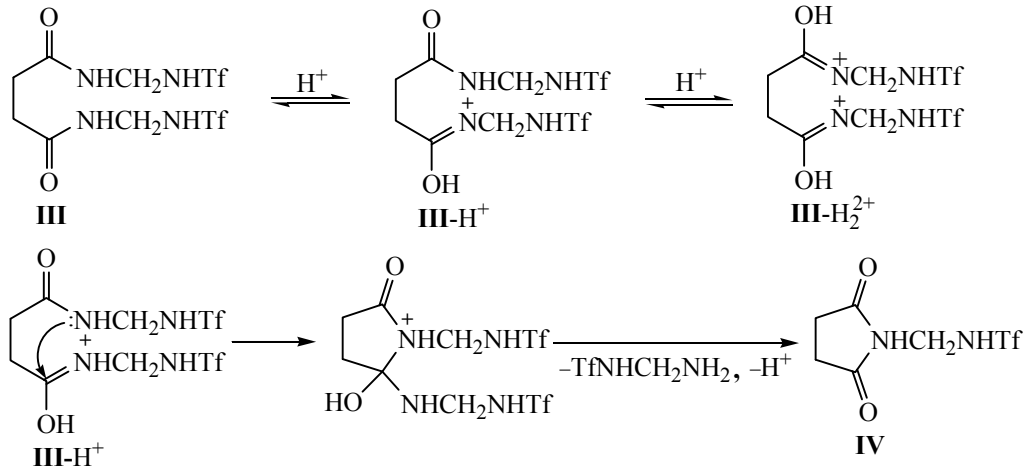
$^{13}C$ NMR spectrum. In the spectrum of compound **IV** a spin-spin splitting of the signal from the fragment $CH_2NH$ was not observed.

The reaction product **IV** might originate from triflamidomethylation of the succinimide formed by a partial hydrolysis and dehydration of succinamide. However this cyclization is hardly possible at room temperature, moreover in ethyl acetate and in the presence of sulfuric acid, namely, in less acid medium than sulfuric acid proper. Besides this mechanism does not account for the pronounced dependence of the reaction direction (Scheme 1) on the nature of the reaction medium: of the presence of insignificant additive of compound **IV** in the reaction along the *a* pathway and of compound **III** as an insignificant impurity in the reaction by the *b* pathway. We tested the possibility of succinamide cyclization under these reaction conditions (Scheme 1), and it was found that at its heating in ethyl acetate with sulfuric acid for 24 h (~40°C) the succinimide actually did not form, as showed the absence of succinimide signals in the $^1H$ and $^{13}C$ NMR spectra of the isolated reaction product. However under these conditions the succinamide suffered transformations, since in the $^1H$ NMR spectrum of the product instead of a singlet of the $CH_2CH_2$ group at 2.26 ppm appeared two triplets at 2.32 and 2.44 ppm (*J* 6.5 Hz), two broadened singlets of NH at 6.8 and 7.3 ppm, and signals of an ethyl group at 4.02 q and 1.16 t ppm. In the $^{13}C$ NMR spectrum signals are observed from two C=O groups at 172.41 and 172.84 ppm, two signals from nonequivalent methylene groups of the fragment $CH_2CH_2$ at 28.84 and 20.52 ppm, and ethyl group signals at 14.06 and 59.75 ppm. These spectra correspond to the formation of ethyl 4-amino-4-oxobutanoate $C_2H_5OOCCH_2CH_2CONH_2$ due to a partial

**Scheme 1.**




**Scheme 2.**



alcoholysis of the succinamide.

We examined the behavior of bis[(trifluoromethylsulfonyl)aminomethyl]succinamide (**III**) under the reaction conditions (Scheme 1) and found that at weak heating for 16 h in contrast to succinamide it nearly totally underwent cyclization (to ~90%) in *N*-[(trifluoromethylsulfonyl)aminomethyl]succinimide (**IV**)as indicated by a coincidence of the NMR signals of the obtained reaction product with the signals of the authentic compound **IV**. This fact suggests the following mechanism of compound **IV** formation.

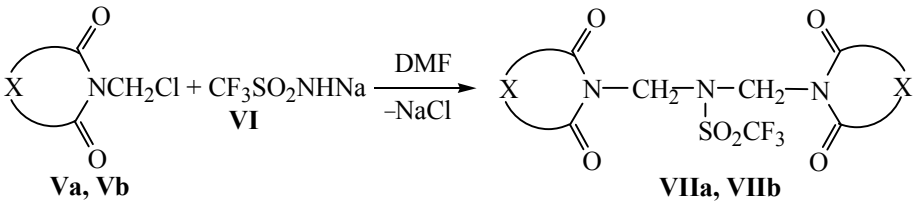
Inasmuch as the carboxylic acids amides are protonated at the oxygen atom (see [4] and references therein) compound **III** in acid medium would exist as an equilibrium mixture of mono- and diprotonated forms (**III**-$H^+$) and (**III**-$H_2^{2+}$).

In the pure sulfuric acid (pathway *a*, Scheme 1) the basicity of both amide nitrogen atoms in the linear compound **III** is suppressed by protonation to (**III**-$H_2^{2+}$), and the reaction is stopped at the stage of its formation. Under less acid conditions (pathway *b*, Scheme 1) the relative concentration of the form (**III**-$H^+$) grows, and the probability of an attack of the nitrogen from the free amide group on the carbon atom of the protonated amide group with the ring closure increases (Scheme 2). The difference in the behavior of succinamide and its disubstituted derivative **III** under the same conditions is due to the greater nucleofuge ability of the $TfNHCH_2NH$ group compared to the $NH_2$ group owing to the strong electron-withdrawing effect of the trifluoromethanesulfonyl group.

In order to increase the yield of compound **III** we studied various versions of this reaction: the condensation in 2-propanol in the presence of potassium carbonate, in dilute and concentrated hydrochloric acid and in 2-propanol in the presence of hydrochloric acid, the condensation in sulfuric acid of triflamide with *N,N*-bis(dihydroxymethylene)-succinamide obtained by the reaction of formaldehyde and succinamide in the presence of potassium carbonate, and also the condensation of oxymethyl derivative of triflamide $CF_3SO_2NHCH_2OH$ with succinamide. However these attempts were unsuccessful.

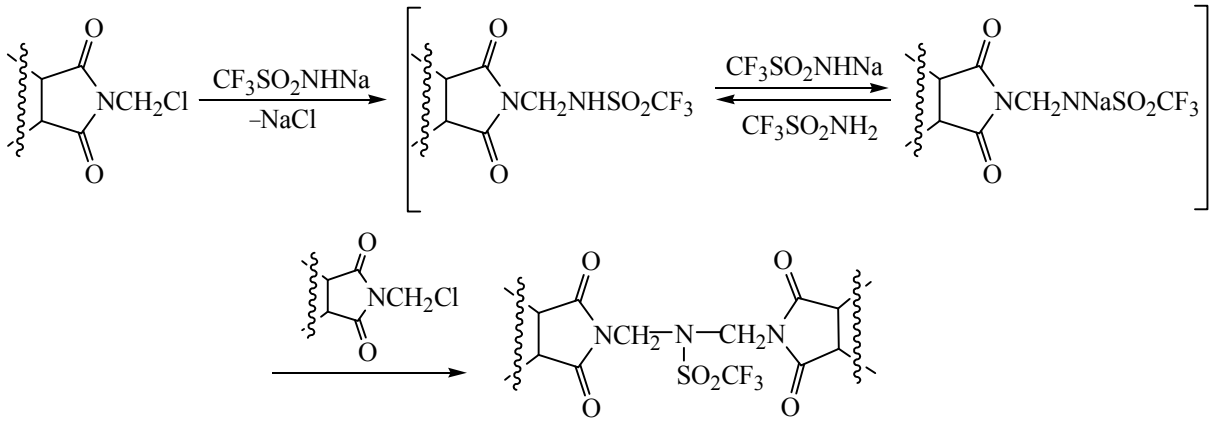
An alternative method for preparation of compound **IV** and its analog, *N*-[(trifluoro-methylsulfonyl)aminomethyl]phthalimide, by the reaction of triflamidesodium salt with N-chloromethyl derivatives of imides was tested by examples of succinimide and phthalimide. N-Oxymethyl derivatives of succinimide and phthalimide obtained in the reaction with formaldehyde water solution were

**Scheme 3.**




X = $CH_2CH_2$ (**a**), 1,2-$C_6H_4$ (**b**).

**Scheme 4.**



chlorinated with $PCl_5$, and thus prepared *N*-chloromethylsuccinimide (**Va**) and *N*-chloromethylphthalimide (**Vb**) were brought into the reaction with triflamide sodium salt (**VI**) in DMF. The result was unexpected: In both cases formed bis-substituted triflamides **VIIa** and **VIIb** (Scheme 3).

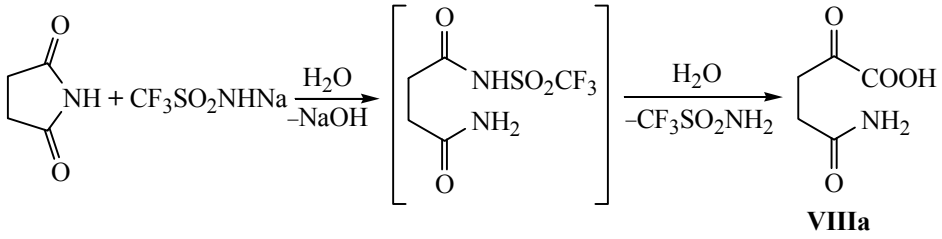
Compound **VIIa** formed as the only reaction product even at the equimolar reagents ratio; the characteristic doublet signal of $NCH_2$ at 4.8 ppm belonging to the expected intermediate compound **IV** did not appear in the $^1$H NMR spectrum of the reaction mixture. It may mean that the intermediately formed monosubstituted product **IV** reacted with *N*-chloromethylsuccinimide far faster than the triflamide sodium salt. It evidently depends on its higher NH-acidity and consequently on the shift of the equilibrium to the right (Scheme 4).

The position of this equilibrium may be estimated by an example of the phthalimide derivative while comparing the values of p$K_a$ of acetic acid and phthalimidoacetic acid equal 4.75 and 3.0 respectively [5]. The latter value is virtually equal to the p$K_a$ of bromoacetic acid, therefore the σ* value of the heterocyclic substituent amounts to ~0.5. The value $\Delta pK_a$ 1.75 corresponds to the content of the sodium salt of compound **IV** >98% , and of triflamide sodium salt <2% manifesting the reason of the experimental result.

We also investigated the ring opening in the succinimide and the phthalimide molecules in the reaction with the triflamide sodium salt in water or in alcohol. The reaction of succinimide in water with excess salt **VI** actually resulted in the ring opening, but the only product of this reaction isolated by the workup of the reaction mixture followed by recrystallization proved to be succinamic acid **VIIIa**.

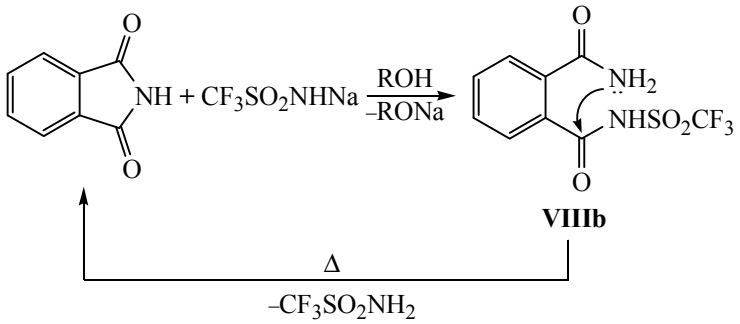
The formation of compound **VIIIa** was proved by NMR spectra, elemental analysis, and the coincidence of the melting point with the published finding. The $^1$H NMR spectrum contains two triplets corresponding to two nonequivalent $CH_2C(O)$ fragments, and two signals of the nonequivalent protons of the amide group $CONH_2$, and in the $^{13}$C NMR spectrum two signals from $CH_2$ and two signals of carbonyl groups are observed. Inasmuch as the succinimide proper does not react with water even at boiling, the reaction evidently proceeds by the type of nucleophilic catalysis with the intermediate formation of N-trifluoromethylsulfonylsuccinamide with a highly nicleofugic group $CF_3SO_2NH$ that is easily hydrolyzed by the liberated alkali. We formerly observed this type nucleophilic catalysis with the elimination of the residue $CF_3SO_2NH$ in the form of trifluoromethanesulfonamide in the reaction of 2-phenyl-2*H*-1,2,3-triazole-

**Scheme 5.**




**VIIIa**

**Scheme 6.**




**VIIIb**

4-carboxamide with acid chloride $CF_3SO_2Cl$ in methanol [6].

The opening of the imide ring occurred also in the phthalimide molecule in the reaction with the triflamide sodium salt in the mixture of methanol with 2-propanol. In the $^1H$ NMR spectrum of the crude reaction product **VIIIb** containing a considerable amount of phthalimide seven signals of equal intensity are observed: two doublets and two triplets of four nonequivalent aromatic protons, two nonequivalent signals of amide group $CONH_2$ in the region ~7.2 ppm. and the signal at 9.1 ppm, i.e., in the region characteristic of the $CF_3SO_2NH$ group. In the $^1H$ NMR spectrum of the proper phthalimide the resonance of all aromatic protons appeared as a singlet at 7.76 ppm. Therefore it was possible to ascribe to product **VIIIb** the structure of *N*-(trifluoromethyl-sulfonyl)phthalamide.

Regretfully, we failed to isolate compound **VIIIb** in the pure state for the recrystallization from 2-propanol of the crude product containing compound **VIIIb** and phthalimide in the ratio ~3:2 resulted in the increased content of phthalimide to the ratio ~1:2, and on the recrystallization from benzene separated a mixture of pure phthalimide and triflamide. It means that at heating of compound **VIIIb** the reverse cyclization easily occurred with the elimination of the triflamide.

## EXPERIMENTAL

IR spectrum of incomplete internal reflection of compound **VIIIa** was recorded on a spectrophotometer Varian 3100 FT-IR. NMR spectra were registered on a spectrometer Bruker DPX-400 at operating frequencies 400 ($^1H$), 100 ($^{13}C$), 376 MHz ($^{19}F$), the chemical shifts were measured relative to TMS ($^1H$, $^{13}C$) and $CCl_3F$ ($^{19}F$). Mass spectrum of electron ionization (70 eV) was obtained on an instrument GCMS-QP5050A Shimadzu (quadrupol mass analyzer) in the direct admission mode. The reaction progress was monitored by TLC on plates with silica gel 60 F-254, eluent hexane–ether, 1:2.

***N,N'*-Bis[(trifluoromethylsulfonyl)aminomethyl]-succinamide (III).** To a solution of 2 g of succinamide pn 55 ml of concn. $H_2SO_4$ was added 6.4 g of triflamide, 1.29 g of paraformaldehyde, and the mixture was stirred for 2 days at room temperature. The reaction mixture was poured on ice with NaCl, the separated precipitate of bis(trifluoromethanesulfonamido)methane (**II**), was filtered off, the water solution was extracted with ether (4× 40 ml), the extract was dried with $MgSO_4$, the solvent was removed. The obtained liquid residue was treated with a mixture ether–hexane, 1:6. The unreacted triflamide and bis(trifluoro-methanesulfonamido)methane (**II**) were soluble in this mixture and were removed; from the residue after recrystallization from dichloromethane the target product was isolated as colorless crystals, mp 185–190°C. $^1H$ NMR spectrum (DMSO-$d_6$), δ, ppm: 2.36 s (4H, $CH_2$), 4.45 d (4H, $NCH_2N$, *J* 6.0 Hz), 8.82 t (2H, NHCO, *J* 6 Hz), 10.13 br.s (2H, $NHSO_2$). $^{13}C$ NMR spectrum (DMSO-$d_6$), δ, ppm: 29.83 s ($CH_2CH_2$), 47.82 (NCN), 119.42 q ($CF_3$, *J* 321.8 Hz), 171.97 s (C=O). $^{19}F$ NMR spectrum ($CD_3CN$), δ, ppm: –79.41. Found, %: C 21.87; H 2.95; N 12.50; S 14.26. $C_8H_{12}F_6N_4O_6S_2$. Calculated, %: C 21.92; H 2.76; N 12.78; S 14.63.

***N*-[(Trifluoromethyl-sulfonyl)aminomethyl]-succinimide (IV)**. To a dispersion of 6.4 g of triflamide, 2 g of succinamide, and 1.29 g of paraformaldehyde in 35 ml of ethyl acetate was added dropwise within 35 min 12 ml of concn. $H_2SO_4$. The reaction mixture was stirred for 4 h at room temperature, then poured on ice with NaCl, extracted thrice with ethyl acetate, the extract was dried with $MgSO_4$, the solvent was removed. The obtained liquid residue was treated with a mixture ether–hexane, 1:6, the insoluble solid residue was filtered off and recrystallized from chloroform to obtain colorless crystals, mp 105–107°C. $^1H$ NMR spectrum (DMSO-$d_6$), δ, ppm: 2.68 s (4H, $CH_2$), 4.75 s (2H, $NCH_2N$), 10.55 br.s (1H, NH). $^{13}C$ NMR spectrum (DMSO-$d_6$), δ, ppm: 27.88 s ($CH_2CH_2$), 45.60 (NCN), 119.57 q ($CF_3$, *J* 320.9 Hz), 176.29 s (CO). $^{19}F$ NMR spectrum ($CD_3CN$), δ, ppm: –74.95. Mass spectrum, *m/z* ($I_{rel}$, %): 191 (6) [*M* – $CF_3$], 162 (9) [$CF_3SO_2NHCH_2$], 127 (79) [*M* – $SO_2CF_3$], 112 (51) [*M* – $NHSO_2CF_3$], 100 (42) [127 – HCN], 84 (38) [112 – CO], 78 (21) [$NSO_2$], 69 (93) [$CF_3$], 56 (84) [$C_3H_4O$], 55 (100) [$C_3H_3O$]. Found, %: C 27.23; H 2.61; F 22.27; N 10.16; S 12.81. $C_6H_7F_3N_2O_4S$. Calculated, %: C 27.70; H 2.71; F 21.91; N 10.77; S 12.32.

***N,N*-Bis(succinimidomethyl)trifluoromethane-sulfonamide (VIIa)**. *N*-Hydroxymethylsuccinimide, obtained by reacting the succinimide with water solution of formaldehyde in the presence of potassium carbonate, was converted into *N*-chloromethylsuccinimide (**Va**) by the reaction with $PCl_5$ in chloroform by procedure [7]. To a solution of 2.54 g (15 mmol) of $CF_3SO_2NHNa$ (**VI**) in 13 ml of DMF was added 2.26 g (0.015 mmol) of *N*-chloromethylsuccinimide, the mixture was stirred for

5 h at room temperature and 5 h more at 60°C, DMF was distilled off in a vacuum, the liquid residue was mixed with 2-propanol, and on cooling from the mixture separated dark-red precipitate. After double recrystallization from 2-propanol yield 0.83 g (15%), mp 166–168°C. $^1$H NMR spectrum (DMSO-$d_6$), δ, ppm: 2.68 s (8H, $CH_2CH_2$), 5.20 s (4H, $NCH_2N$). $^{13}$C NMR spectrum (DMSO-$d_6$), δ, ppm: 27.95 s ($CH_2CH_2$), 52.99 (NCN), 118.98 q ($CF_3$, *J* 323.6 Hz), 176.94 s (CO). $^{19}$F NMR spectrum (DMSO-$d_6$), δ, ppm: –75.14. Found, %: C 35.55; H 3.38; F 16.39; N 11.03; S 8.97. $C_{11}H_{12}F_3N_4O_6S$.. Calculated, %: C 35.58; H 3.26; F 15.35; N 11.32; S 8.64.

***N,N*-Bis(phthalimidomethyl)trifluoromethanesulfonamide (VIIb)**. *N*-Hydroxymethylphthalimide, obtained by reacting the phthalimide with water solution of formaldehyde in the presence of potassium carbonate [8], was converted into *N*-chloromethylphthalimide (**Vb**) by the reaction with $PCl_5$ in chloroform by procedure [9]. To a solution of 0.96 g (5.6 mmol) of $CF_3SO_2NHNa$ (**VI**) in 4 ml of DMF was added by small portions 1 g (5.1 mmol) of N-chloromethylenephthalimide, the mixture was stirred for 2 h at room temperature, 4 h at 80°C, 5 h at 110°C, and 5 h at 140°C, then it was poured on ice with NaCl, the separated precipitate was filtered off and recrystallized from acetone. Yield 0.9 g (38%), mp 197–199°C. $^1$H NMR spectrum (DMSO-$d_6$), δ, ppm: 5.50 s (4H, $CH_2$), 7.92 m (8H, $H_{arom}$). $^{13}$C NMR spectrum (DMSO-$d_6$), δ, ppm: 52.89 ($CH_2$), 119.11 q ($CF_3$, *J* 323.8 Hz), 123.70 ($C^{4,7}$), 131.25 ($C^{8,9}$), 135.12 ($CH^{5,6}$), 166.96 (CO). $^{19}$F NMR spectrum (DMSO-$d_6$), δ, ppm: –75.47. Found, %: C 48.83; H 2.89; F 12.19; N 8.99; S 6.80. $C_{19}H_{12}F_3N_3O_6S$. Calculated, %: C 48.62; H 3.01; F 12.14; N 8.95; S 6.84.

**4-Amino-4-oxobutanoic acid (VIIIa)**. To a solution of 0.5 g (5 mmol) of succinimide in 7 ml of water was added by small portions 1.88 g (11 mmol) of triflamide sodium salt. The reaction mixture was heated for 6 h at 90°C, water was evaporated on a rotary evaporator at a reduced pressure. The obtained glassy residue (2 g) was treated with ethyl acetate removing the soluble in this solvent unreacted triflamide sodium salt. Insoluble dark-red precipitate (1.1 g) was treated with 5 ml of 5% HCl, thoroughly stirred, evaporated at a reduced pressure, the residue was recrystallized from 2-propanol. Yield 0.55 g (44% ), mp 158–160°C (mp 157°C [10]). IR spectrum, ν, cm$^{-1}$: 3364, 3203 (N–H), 2933 (C–H), 2770–2470 (OH), 1726, 1704 (COOH), 1644 ($CONH_2$), 1581, 1414, 1359, 1269, 1201, 1170, 1125, 966, 923, 797, 660, 602. $^1$H NMR spectrum (DMSO-$d_6$), δ, ppm: 2.28 t (2H, $CH_2COOH$, *J* 6.6 Hz), 2.39 t (2H, $CH_2CONH_2$, *J* 6.6 Hz), 6.76 s (1H, $CONH_A$), 7.29 s (1H, $CONH_B$), 12.05 br.s (1H, OH). $^{13}$C NMR spectrum (DMSO-$d_6$), δ, ppm: 29.00 ($CH_2COOH$), 29.70 ($CH_2CONH_2$), 173.10 ($CONH_2$), 173.93 (COOH). Found, %: C 40.90; H 6.01; N 11.32. $C_4H_7NO_3$..Calculated, %: C 41.03; H 6.03; N 11.96.

***N*-(Trifluoromethylsulfonyl)phthalamide (VIIIb)**. To a dispersion of 0.5 g (3 mmol) of phthalimide in a mixture of 10 ml of 2-propanol and 2 ml of MeOH was added by small portions 1.4 g (8 mmol) of triflamide sodium salt. The reaction mixture was heated for 16 h at 75°C, then 5 ml of 5% HCl was added, the mixture was thoroughly stirred, the solvents were evaporated at a reduced pressure. In the residue (1.7 g, mp 177°C) according to $^1$H NMR data, ~40% phthalimide was present. $^1$H NMR spectrum (DMSO-$d_6$), δ, ppm (phthalimide signals are omitted): 7.17 s (1H, $CONH_A$), 7.28 t (1H, $H_\beta$, *J* 7.1 Hz), 7.35 t (1H, $H'_\beta$ *J* 7.2 Hz), 7.48 d (1H, $H_\alpha$, *J* 7.5 Hz), 7.60 d (1H, $H'_\alpha$, *J* 7.7 Hz), 9.14 s (1H, $NHSO_2$). The signal of proton $CONH_B$ overlapped with triplet signals of protons $H_\beta$ and $H'_\beta$ as showed the integral intensity of this spectral region overestimated by one proton. $^{13}$C NMR spectrum (DMSO-$d_6$), δ, ppm: 124.06, 126.14, 127.41, 128.89, 129.82, 139.06, 171.04, 173.62. We failed to accumulate the signal of $CF_3$ group. In the $^{19}$F NMR spectrum of samples obtained after removal of solvent from the reaction mixture and recrystallization of the residue from 2-propanol only the signal of trifluoromethanesulfonamide was present.

The attempts to purify the reaction product by recrystallization from 2-propanol or benzene resulted in its reverse cyclization into phthalimide with the triflamide elimination (see the text).

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