

## Trifluoromethanesulfonic Hydrazides

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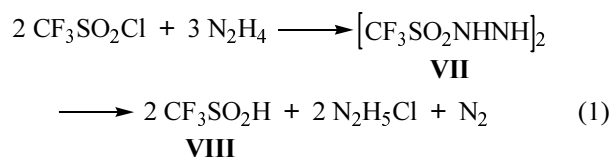
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**Abstract**— We succeeded to observe at low temperature in reactions of trifluoromethanesulfonic anhydride and trifluoromethanesulfonyl chloride with hydrazine, phenyl hydrazine, and 1,1-dimethylhydrazine a formation of the corresponding trifluoromethanesulfonic hydrazides that at heating to room temperature decomposed liberating nitrogen and affording trifluoromethanesulfonic acid. 2-Phenyl-2*H*-1,2,3-triazole-4-carboxylic hydrazide reacted with trifluoromethanesulfonic anhydride to furnish trifluoro-*N'*-(2-phenyl-2*H*-1,2,3-triazol-4-ylcarbonyl)methanesulfonic hydrazide that decomposed at heating with elimination of trifluoromethanesulfonic acid and nitrogen yielding 2-phenyl-2*H*-1,2,3-triazole-4-carbaldehyde.

Nitrogen derivatives of pentafluoroalkanesulfonic acids, like amides  $R_FSO_2NH_2$  and their *N*-substituted derivatives, or azides  $R_FSO_2N_3$  are successfully used in the organic synthesis [1–5]. At the same time the published information on perfluoroalkanesulfonic hydrazides is relatively scanty. It was demonstrated that perfluoroalkenesulfonyl halides were reduced with hydrazine into perfluoroalkanesulfonic acids derivatives [6–10] but in neither of these studies the intermediate hydrazides  $R_FSO_2NHNH_2$  were isolated as individual compounds or at least characterized. Among the *N*-substituted hydrazides  $R_FSO_2NHNH_X$  only functionally-substituted trifluoromethanesulfonic acid hydrazides  $CF_3SO_2NHNH_X$  ( $X = PhCO, t-BuOCO$ ) are known that are used in oxidation of alkyl halides to the corresponding hydrazones of aldehydes and ketones [11]; therewith the triflate group is also reduced to triflinate one. No data were published on reactions between perfluoroalkanesulfonic acids derivatives with alkyl- or arylhydrazines.

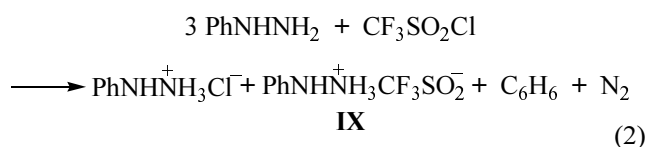
In this study we investigated reactions of hydrazine (**I**), phenyl hydrazine (**II**), 1,1-dimethylhydrazine (**III**), and 2-phenyl-2*H*-1,2,3-triazole-4-carboxylic hydrazide (**IV**) with trifluoromethanesulfonyl chloride  $CF_3SO_2Cl$  (**V**), and/or trifluoromethanesulfonic anhydride  $(CF_3SO_2)_2O$  (**VI**) aiming at preparation of the corresponding trifluoromethanesulfonic hydrazides and their characterization. Acyl chloride **V** reacted with excess hydrazine (**I**) in anhydrous THF at  $-50^\circ C$  without nitrogen liberation, and in the  $^{13}C$  NMR spectrum recorded at  $-30^\circ C$  a quartet of  $CF_3$  group was observed

at 122 ppm with a coupling constant  $^1J_{CF}$  322.8 Hz. The signal belonged to the trifluoromethanesulfonic hydrazide  $CF_3SO_2NHNH_2$  (**VII**). The heating to room temperature was accompanied with a vigorous nitrogen evolution, and in the  $^{13}C$  NMR spectrum a quartet appeared at 125–126 ppm with a coupling constant  $^1J_{CF}$  350–360 Hz (depending on the solvent). The signal belonged to the  $CF_3$  group of trifluoromethanesulfonic acid or its salt. The signal in the  $^{19}F$  NMR spectrum is shifted at the same time upfield from  $-78$  to  $-86$  ppm. Inasmuch as all trifluoromethanesulfonic acid derivatives  $CF_3SO_2X$  ( $X = OH, OSO_2CF_3, Cl, NH_2, NR_2, NHSO_2CF_3, N_3$ ) regardless of *X* character possess signals with a definite position in the  $^{13}C$  and  $^{19}F$  NMR spectra (119...122 and  $-78$ ... $-79$  ppm respectively), and the corresponding coupling constants  $^1J_{CF}$  are 319–321 Hz the resonances arising at heating can be unambiguously ascribed to trifluoromethanesulfonic acid derivatives. Thus the hydrazine reaction with acyl chloride **V** proceeded along equation (1) with elimination of  $CF_3SO_2H$  molecule from the intermediate hydrazide **VII** yielding either free acid **VIII**, or at large hydrazine excess its salt  $N_2H_5^+CF_3SO_2^-$ .



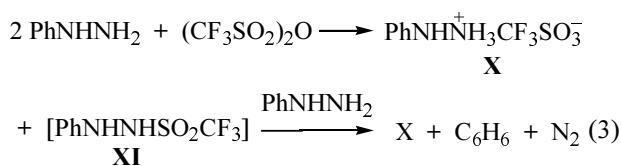
The above spectral criteria of distinction between triflate or triflinate derivatives were applied to

investigation of reactions between trifluoromethanesulfonic anhydride and trifluoromethanesulfonyl chloride with substituted hydrazines **II** and **III**. The reaction of phenylhydrazine (**II**) with acyl chloride **V** was carried out under various conditions: without solvent, in dichloromethane, carbon tetrachloride, or in DMSO-*d*<sub>6</sub> directly in an NMR tube at the reagents ratio from 1 : 1 to 2 : 1, at -78°C and room temperature. In all cases the reaction proceeded according to equation:



Benzene formation was revealed by appearance of a singlet at 7.33 ppm in the <sup>1</sup>H NMR spectrum and of a singlet at 129.89 ppm in the <sup>13</sup>C NMR spectrum of the reaction mixture. In contrast to hydrochloride 2-phenylhydrazinium triflate (**IX**) is soluble in ether, and thus these two salts may be separated. The structure of salt **IX** is confirmed by the presence in its <sup>13</sup>C NMR spectrum alongside the signals from the benzene ring also a quartet at 125 ppm with a coupling constant <sup>1</sup>J<sub>CF</sub> 350–360 Hz (depending on the solvent), and by a singlet at -86.04 ppm in its <sup>19</sup>F NMR spectrum; the composition of the salt was proved by elemental analysis. The triflates are additionally distinguished from triflinates by the absorption bands in the IR spectra corresponding to the stretching vibrations of the SO<sub>2</sub> groups: In the IR spectra of all triflates appear strong bands of symmetric (1220–1160 cm<sup>-1</sup>) and antisymmetric (1440–1357 cm<sup>-1</sup>) vibrations of SO<sub>2</sub> group. In the spectra of triflinates only a band in the 1010–1150 cm<sup>-1</sup> region is observed [6, 12, 13]. The lack of the high-frequency band in the IR spectrum of salt **IX** confirms its triflinate structure.

The reaction of phenylhydrazine with trifluoromethanesulfonic anhydride (**VI**) in dichloromethane afforded as the main product phenylhydrazinium triflate (**X**) identical to a sample obtained directly from phenylhydrazine and trifluoromethanesulfonic acid.



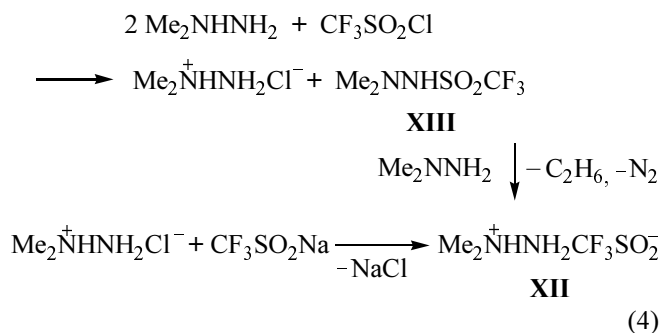
The CF<sub>3</sub> quartet in the <sup>13</sup>C NMR spectrum of salt **X** appears in the region characteristic of triflates, at

121.10 ppm with a coupling constant <sup>1</sup>J<sub>CF</sub> 321.8 Hz, and the signal in the <sup>19</sup>F NMR spectrum is observed at -77.76 ppm. In the IR spectrum of salt **X** the very strong band of stretching vibrations of SO<sub>2</sub> group is present which lacks in the IR spectrum of salt **IX**. The <sup>19</sup>F NMR spectrum of the residue after separation of salt **X** contains a main signal at -85.54 ppm belonging to salt **IX** (50%), and also two signals in the “triflate” region at -77.75 ppm (30%) and -75.59 ppm (20%). The first one corresponds to salt **X**, whereas the second belongs presumably to an intermediate trifluoromethanesulfonic *N*-phenylhydrazide (**XI**). Benzenesulfonic *N*-phenylhydrazide PhSO<sub>2</sub>NHNHPh is known to eliminate benzenesulfonic acid at treatment with alkali yielding phenyldiimide C<sub>6</sub>H<sub>5</sub>N=NH that decomposes further into benzene and nitrogen [14]. Apparently the *N*-phenylhydrazide of the stronger trifluoromethanesulfonic acid suffers spontaneous decomposition.

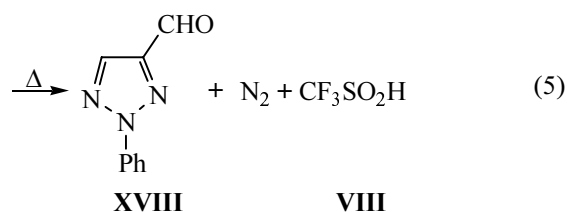
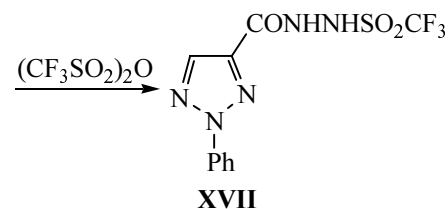
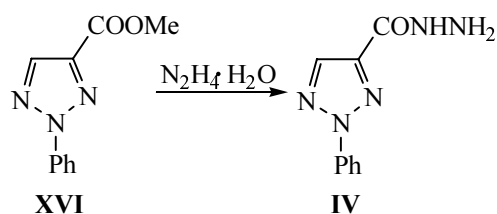
The reaction of acyl chloride **V** with 1,1-dimethylhydrazine (**III**) in anhydrous THF at room temperature also occurred with nitrogen evolution. The <sup>13</sup>C NMR spectra of reaction mixtures contain two quartets of CF<sub>3</sub> groups: at 121 ppm with a coupling constant <sup>1</sup>J<sub>CF</sub> 322 Hz, and at 125 ppm with a coupling constant <sup>1</sup>J<sub>CF</sub> 356 Hz, and also two methyl group signals at 34 and 47 ppm. In the <sup>1</sup>H NMR spectrum the corresponding singlets from NMe groups appear at 2.5 and 2.8 ppm. The 47 ppm resonance in the <sup>13</sup>C NMR spectrum and the respective signal at 2.8 ppm in the <sup>1</sup>H NMR spectrum belong to methyl groups in the 1,1-dimethylhydrazinium ion [Me<sub>2</sub>NHNH<sub>2</sub>]<sup>+</sup> as prove the <sup>13</sup>C NMR spectra of 1,1-dimethylhydrazinium chloride, triflate, and triflinate that have been independently prepared by treating dimethylhydrazine with hydrochloric or trifluoromethanesulfonic acid, or from 1,1-dimethylhydrazinium chloride and sodium triflinate respectively. In the IR spectrum of 2,2-dimethylhydrazinium triflinate (**XII**) lacks the vibration band ν<sub>as</sub> SO in the region 1440–1357 cm<sup>-1</sup> as has been observed in the spectrum of salt **IX**.

In reactions carried out below -30°C no nitrogen evolution occurred, and in the <sup>13</sup>C NMR of the reaction mixture appeared only signals at 34 and 120 ppm from trifluoromethanesulfonic 2,2-dimethylhydrazide CF<sub>3</sub>SO<sub>2</sub>NHN(CH<sub>3</sub>)<sub>2</sub> (**XIII**). In the <sup>15</sup>N NMR spectrum the resonances from this compound are observed at -286.4 (NH) and -356.9 ppm (NMe<sub>2</sub>), and in the <sup>1</sup>H NMR spectrum is present only the signal of non-protonated methyl group at 2.5 ppm.

The cleavage of trifluoromethanesulfonic acid from hydrazide **XIII** in contrast to hydrazides **VII** and **XI**



can proceed solely as 1,1-elimination affording dimethylaminonitrene ( $\text{Me}_2\text{NN}:$ ) that should either dimerize into tetramethyltetrazene or decompose into ethane and nitrogen [15]. The vigorous evolution of gaseous products and triflate formation at heating the reaction mixture to room temperature confirms that the reaction occurs in conformity to equation (4). However the concomitant formation of tetramethyltetrazene  $\text{Me}_2\text{NN}=\text{NNMe}_2$  or its salts cannot be excluded. In some runs on treating the evaporated reaction mixture with methanol we observed additional signals in the NMR spectra at the following chemical shifts: 3.3 ppm ( $^1\text{H}$ ), 57 ppm ( $^{13}\text{C}$ ), and 69.8 and  $-230.0$  ppm ( $^{15}\text{N}$ ). These signals may be assigned to tetramethyltetrazene salt. To clear out this possibility we synthesized along procedure [16] tetramethyltetrazene (XIV), prepared its triflate salt XV, and recorded  $^1\text{H}$ ,  $^{13}\text{C}$ , and  $^{15}\text{N}$  spectra of both compounds. In the NMR spectra of tetramethyltetrazene recorded in  $\text{DMSO}-d_6$  were observed the following signals: 2.73 ppm ( $^1\text{H}$ ), 40.83 ppm ( $^{13}\text{C}$ ), and two signals in the  $^{15}\text{N}$  NMR spectrum in the regions characteristic of NMe and N=N groups. In the  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra of salt XV prepared *in situ* by adding trifluoromethanesulfonic acid to a solution of compound XIV in  $\text{DMSO}-d_6$  directly in an NMR tube the corresponding signals underwent downfield shifts to 3.08 and 42.29 ppm respectively. These results showed that no tetrazene XIV or its salt XV formed in reaction (4). The signals at 3.3 ppm ( $^1\text{H}$ ) and 57 ppm ( $^{13}\text{C}$ ) were alternatively suggested to belong to methylation product of the intermediate hydrazide XIII, 1,1,1-trimethyl-2-[(trifluoromethyl)sulfonyl]hydrazinium cation  $\text{CF}_3\text{SO}_2\text{NHN}^+\text{Me}_3$ . This assignment is confirmed by coincidence of these chemical shifts with the known data on  $^1\text{H}$ ,  $^{13}\text{C}$ , and  $^{15}\text{N}$  spectra of 1,1,1-trimethylhydrazonium salts [17]. Inasmuch as the esters of sulfonic acids were known to be able to behave as alkylating agents and taking into account that the signals of 1,1,1-trimethyl-2-[(trifluoromethyl)sulfonyl]hydrazinium cation were observed only if the reaction mixture was treated with methanol it was presumable that the



cation formed through alkylation of hydrazide XIII with intermediate methyl trifluoromethylsulfonate.

We expected that 2-phenyl-2*H*-1,2,3-triazole-4-carboxylic hydrazide (IV) which we had prepared by treating methyl 2-phenyl-2*H*-1,2,3-triazole-4-carboxylate (XVI) with hydrazine hydrate would react with trifluoromethanesulfonic anhydride or trifluoromethanesulfonyl chloride similarly to the described reactions of functionally-substituted hydrazides  $\text{XNHNH}_2$  ( $\text{X} = \text{PhCO}$ ,  $t\text{-BuOCO}$ ) [11] affording trifluoro-*N*-(2-phenyl-2*H*-1,2,3-triazol-4-ylcarbonyl)methanesulfonic hydrazide (XVII). Actually the mixed hydrazide XVII proved to be more stable than the above mentioned hydrazides VII, XI, and XIII lacking electron-withdrawing groups, and it was prepared by reaction between compound IV and anhydride VI. However at the attempt to purify hydrazide XVII by sublimation it suffered thermal degradation with nitrogen evolution and formation of 2-phenyl-2*H*-1,2,3-triazole-4-carbaldehyde (XVIII) and trifluoromethanesulfonic acid (VIII).

The formation of aldehyde XVIII was proved by chromatography with an authentic sample. In the  $^1\text{H}$  NMR spectrum appeared singlets at 9.13 and 10.20 ppm in 1:1 ratio belonging to trifluoromethanesulfonic acid (VIII) and aldehyde XVIII. The singlet of aldehyde proton in the spectrum of authentic aldehyde XVIII is also observed at 10.20 ppm.

## EXPERIMENTAL

IR spectra were recorded on spectrophotometer IKS-29 from thin film on CaF<sub>2</sub> plates or from samples pelletized with KBr. NMR spectra were registered on a spectrometer Bruker DPX-400 at operating frequencies 400 (<sup>1</sup>H), 100 (<sup>13</sup>C), 40 (<sup>15</sup>N), and 376 MHz (<sup>19</sup>F) in DMSO-*d*<sub>6</sub> (if not other indicated), internal reference HMDS, chemical shifts are reported with respect to TMS (<sup>1</sup>H, <sup>13</sup>C), CH<sub>3</sub>NO<sub>2</sub> (<sup>15</sup>N), and CCl<sub>3</sub>F (<sup>19</sup>F). The chemical shifts for <sup>15</sup>N were determined from two-dimensional spectra 2D(<sup>1</sup>H-<sup>15</sup>N) with the use of gradient probe in *hmbcgp* mode.

**Reaction of trifluoromethanesulfonyl chloride with phenylhydrazine** At cooling to -78°C and stirring to a solution of 2.16 g (0.02 mol) of phenylhydrazine in 10 ml of dichloromethane was added dropwise a solution of 1.69 g (0.01 mol) trifluoromethanesulfonyl chloride in 11 ml of dichloromethane. The reaction mixture was stirred at room temperature and stored for 24 h at -12°C, then evaporated, treated with anhydrous ethyl ether, the precipitate of 2-phenylhydrazine hydrochloride (0.96 g, 66%) was separated, washed with anhydrous ether (4×10 ml), the ether and arising benzene was distilled off. We obtained 1.6 g (66%) of 2-phenylhydrazinium triflate (**IX**), mp 78–80°C. IR spectrum,  $\nu$ , cm<sup>-1</sup>: 3200, 3050–2830, 2700, 1600, 1580, 1500, 1150, 1030, 980, 900, 850, 690, 600. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 6.97 t (1H, H<sub>p</sub>), 7.26 d (2H, H<sub>O</sub>), 7.47 t (2H, H<sub>m</sub>), 9.83 br.s (4H, NH). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm: 114.49 (C<sub>O</sub>), 121.67 (C<sub>p</sub>), 125.03 (CF<sub>3</sub>, <sup>1</sup>J<sub>CF</sub> 356.0 Hz), 129.07 (C<sub>m</sub>), 145.52 (C<sup>1</sup>). <sup>19</sup>F NMR spectrum,  $\delta$ , ppm: -86.04. Found, %: C 34.31; H 3.84; F 22.82; N 11.22; S 13.85. C<sub>7</sub>H<sub>9</sub>F<sub>3</sub>N<sub>2</sub>O<sub>2</sub>S. Calculated, %: C 34.71; H 3.74; F 23.53; N 11.57; S 13.24.

**Reaction of trifluoromethanesulfonic anhydride with phenylhydrazine.** At cooling to -78°C and stirring to a solution of 2.16 g (0.02 mol) of phenylhydrazine in 10 ml of dichloromethane was added dropwise a solution of 2.82 g (0.01 mol) of trifluoromethanesulfonic anhydride in 10 ml of dichloromethane. The reaction mixture was left standing for 24 h at -12°C, evaporated, treated with anhydrous ethyl ether, the 3.86 g of precipitate was separated, washed with dichloromethane (4×10 ml), and dried in a vacuum. We obtained 2.03 g (79%) of 2-phenylhydrazinium triflate (**X**), mp 160°C. IR spectrum,  $\nu$ , cm<sup>-1</sup>: 3300, 3200–2970, 1600, 1500, 1220, 1160, 1020, 750, 680, 640. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 6.28 br.s (4H, NH), 6.74 t (1H, H<sub>p</sub>), 6.85 d (2H, H<sub>O</sub>), 7.18 t (2H, H<sub>m</sub>). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm: 112.70 (C<sub>O</sub>), 118.74

(C<sub>p</sub>), 120.79 (CF<sub>3</sub>, <sup>1</sup>J<sub>CF</sub> 321.9 Hz), 128.77 (C<sub>m</sub>), 149.78 (C<sup>1</sup>). <sup>19</sup>F NMR spectrum,  $\delta$ , ppm: -77.63. Found, %: C 32.69; H 3.66; F 21.99; N 11.25; S 12.42. C<sub>7</sub>H<sub>9</sub>F<sub>3</sub>N<sub>2</sub>O<sub>3</sub>S. Calculated, %: C 32.56; H 3.51; F 22.07; N 10.85; S 12.42.

**Reaction of trifluoromethanesulfonic acid with phenylhydrazine.** To 1.08 g (0.01 mol) of phenylhydrazine cooled to -78°C was added dropwise a solution of 1.50 g (0.01 mol) of trifluoromethanesulfonic acid in 0.9 ml of distilled water maintaining the temperature of the reaction mixture at -78°C. Then the cooling was removed, and the reaction mixture was left standing for 24 h at -12°C. On evaporation we obtained 2.57 g (quantitative yield) of 2-phenylhydrazinium triflate (**X**), mp 160°C.

**2-Phenyl-2H-1,2,3-triazole-4-carboxylic hydrazide (IV).** A mixture of 3 g (0.015 mol) of methyl 2-phenyl-2H-1,2,3-triazole-4-carboxylate (**XVI**) [18] and 10 ml of hydrazine hydrate was heated at stirring to 110–115°C for 1 h. The separated precipitate was filtered off, washed with water, and dried. We obtained 2.72 g (90.7%) of hydrazide **IV**, mp 181–182°C. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 4.60 br.s (2H, NH<sub>2</sub>), 7.44 t (1H, H<sub>p</sub>), 7.56 t (2H, H<sub>m</sub>), 8.05 d (2H, H<sub>O</sub>), 8.40 s (1H, H<sup>5</sup>), 9.85 br.s (1H, NHCO). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm: 118.73 (C<sub>O</sub>), 128.17 (C<sub>p</sub>), 129.44 (C<sub>m</sub>), 135.70 (C<sup>5</sup>), 138.78 (C<sup>1</sup>), 143.12 (C<sup>4</sup>), 158.52 (C=O). Found, %: C 53.89; H 5.04; N 34.74. C<sub>9</sub>H<sub>9</sub>N<sub>5</sub>O. Calculated, %: C 53.20; H 4.46; N 34.47.

**Trifluoro-N'-(2-phenyl-2H-1,2,3-triazol-4-yl-carbonyl)methanesulfonic hydrazide (XVII).** To a solution of 1.88 g (0.067 mol) of trifluoromethanesulfonic anhydride in 50 ml of dichloromethane at cooling with solid carbon dioxide was added in small portions while stirring 2.71 g (0.0133 mol) of 2-phenyl-2H-1,2,3-triazole-4-carboxylic hydrazide (**IV**). Then the reaction mixture was heated to room temperature (it turned thick in the process). The mixture was stirred for 1 h, the solvent was evaporated, 20 ml of anhydrous ether was added, after stirring the hydrazide **IV** triflate was filtered off, and the filtrate was evaporated to obtain 1.13 g (50%) of compound **XVII**. After column chromatography on silica gel (eluent hexane–ether, 2:1) we obtained colorless crystals, mp 210°C. <sup>1</sup>H NMR spectrum,  $\delta$ , ppm: 7.50 t (1H, H<sub>p</sub>), 7.62 t (2H, H<sub>m</sub>), 8.09 d (2H, H<sub>O</sub>), 8.59 s (1H, H<sup>5</sup>), 11.37 s (1H, NHCO). <sup>13</sup>C NMR spectrum,  $\delta$ , ppm: 119.16 (C<sub>O</sub>), 119.24 (CF<sub>3</sub>, <sup>1</sup>J<sub>CF</sub> 322.6 Hz), 128.90 (C<sub>p</sub>), 129.81 (C<sub>m</sub>), 137.07 (C<sup>5</sup>), 138.72 (C<sup>1</sup>), 141.35 (C<sup>4</sup>), 159.33 (C=O). <sup>19</sup>F NMR spectrum,  $\delta$ , ppm: -76.33. Found, %: C 36.49; H 2.39; F 16.84; N 20.48; S 10.05.

C<sub>10</sub>H<sub>8</sub>F<sub>3</sub>N<sub>5</sub>O<sub>3</sub>S. Calculated, %: C 35.83; H 2.41; F 17.00; N 20.89; S 9.56.

Compound **XVII** (1 g) was heated in a vacuum (1–3 mm Hg) in a device for sublimation in a vacuum. In the cooling trap settled out colorless crystals whose <sup>1</sup>H NMR spectrum was identical to that of an authentic 2-phenyl-2*H*-1,2,3-triazole-4-carbaldehyde (**XVIII**) in CDCl<sub>3</sub>, δ, ppm: 7.44 t (1H, H<sub>p</sub>), 7.53 t (2H, H<sub>m</sub>), 8.13 d (2H, H<sub>o</sub>), 8.25 s (1H, CH), 10.21 s (1H, CHO).

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