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1-Bromo-3,3,3-trifluoro-1-nitropropene: Synthesis and Reaction with Phenyl Azide

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Abstract—An improved procedure was developed for the synthesis of 3,3,3-trifluoro-1-nitropropene, and a new representative of *gem*-bromonitroalkenes, 1-bromo-3,3,3-trifluoro-1-nitropropene, was synthesized therefrom. Its reaction with phenyl azide gave a mixture of two regioisomeric 1,2,3-triazoles, from which pure 5-nitro-1-phenyl-4-(trifluoromethyl)-1*H*-1,2,3-triazole was isolated.

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Chemistry of functionalized unsaturated nitro compounds is an extensively developing basic research line in modern organic chemistry [1–3], and conjugated nitroalkenes are promising synthons and building blocks for the preparation of various organic compounds, including cyclic structures obtainable via cycloaddition reactions [4–7]. Introduction of a trihalomethyl group into the vicinal position with respect to the nitro group considerably extends the synthetic potential of the resulting structures and endows them with a number of practically important properties. It is sufficient to note that derivatives of carbo- and heterocyclic systems containing CF_3, CCl_3, or CBr_3 group exhibit a broad spectrum of biological activity; in particular, trifluoromethyl-substituted pyrrole and cyclohexene derivatives were found to possess antimicrobial and enzymatic activity [8–11].

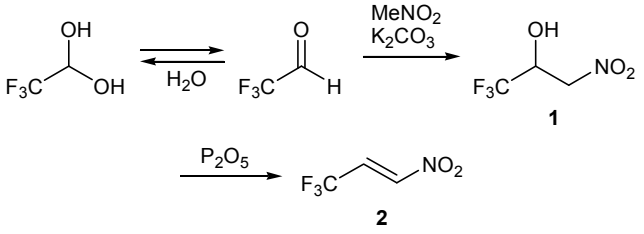
It is also important that 3,3,3-trifluoro-1-nitropropene was used as a starting material to obtain a new class of peptidomimetics containing a stereogenic $CH(CF_3)NH$ group instead of the natural peptide C(=O)NH moiety, which made it possible to eliminate some pharmacological drawbacks of peptides (such as bioavailability and sensitivity to enzymatic hydrolysis) and retain natural peptide properties [12]. The known "fluorine effect" is important in medicinal chemistry for the design of new peptide-based medicinals (peptidomimetics).

In comparison to 3,3,3-trifluoro-1-nitropropene, the synthetic potential of its analog containing a bromine atom in the geminal position with respect to the nitro group seems even more promising. Among 3,3,3-trihalo-1-bromo-1-nitropropenes, only 1-bromo-3,3,3-trichloro-1-nitropropene and 1,3,3,3-tetrabromo-1-nitropropene have been reported so far. 1-Bromo-3,3,3-trichloro-1-nitropropene as the *E* isomer was synthesized by halogenation–dehydrohalogenation of the corresponding nitroalkenes [13, 14]. 1,3,3,3-Tetrabromo-1-nitropropene was recently obtained as a mixture of *Z* and *E* isomers according to a similar procedure [15].

The present work was aimed at developing a procedure for the synthesis of 1-bromo-3,3,3-trifluoro-1-nitropropene. As precursor we used 3,3,3-trifluoro-1-nitropropene (**2**). It was prepared by condensation of nitromethane with trifluoroacetaldehyde hydrate [16] which was synthesized by reduction of ethyl

trifluoroacetate with sodium tetrahydridoborate [17]. The reaction of trifluoroacetaldehyde hydrate with nitromethane afforded 1,1,1-trifluoro-3-nitropropan-2-ol (**1**) (Scheme 1) We succeeded in improving the procedures described in [17, 18] by using ethanol as solvent instead of THF [17] in the synthesis of trifluoroacetaldehyde hydrate and changing the reactant ratio in the reaction of the latter with nitromethane to 1:1 (instead of 1:2 [18]). As a result, the yield of **1** increased from 47 to 94%. Distillation of **1** over phosphoric anhydride afforded (*E*)-3,3,3-trifluoro-1-nitropropene (**2**) in 76% yield.

Scheme 1.



1-Bromo-3,3,3-trifluoro-1-nitropropene (**4**) was synthesized in two steps (Scheme 2). In the first step, nitroalkene **2** was treated with molecular bromine at reduced temperature (–20 to –10°C) for 48 h. Dibromide **3** thus formed was isolated as a mixture of two diastereoisomers **3a** and **3b** at a ratio of 4:1. In the second step, isomer mixture **3a**/**3b** was subjected to dehydrobromination by the action of 2-methylpyridine in diethyl ether. Bromonitroalkene **4** was obtained as a concentrated solution in diethyl ether (yield ~80% according to the ^{1}H NMR data). We failed to isolate compound **4** in the pure state, since it instantaneously decomposed at room temperature. Newly synthesized compounds **3a**/**3b** and **4** are volatile oils and are strong lachrymators.

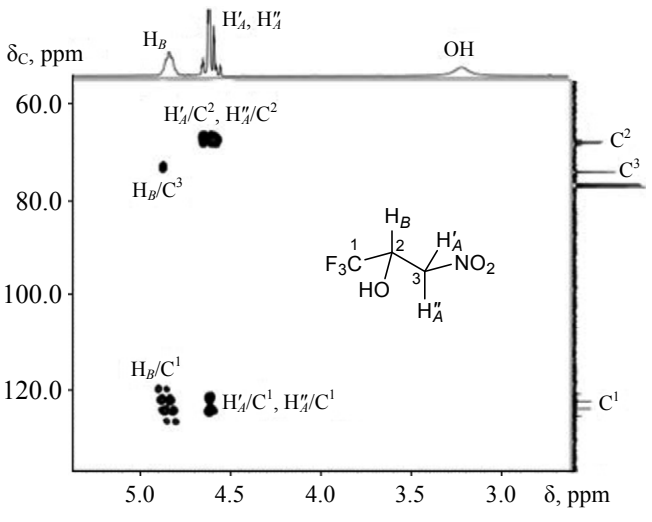
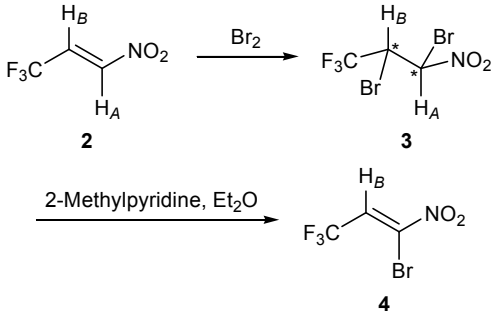


Fig. 1. ^{1}H–^{13}C HMBC spectrum of 1,1,1-trifluoro-3-nitropropan-2-ol (**1**).

Scheme 2.



The structure of **1**–**4** was studied by IR, UV, and NMR (^{1}H, ^{13}C, ^{19}F) spectroscopy, including heteronuclear HMQC and HMBC NMR techniques. The data were compared with those reported previously for structurally related compounds with CCl_3 and CBr_3 groups [15, 19].

The ^{1}H and ^{13}C NMR spectra of **1**–**4** were consistent with the assumed structures. Nitro alcohol **1** displayed in the ^{1}H NMR spectrum two doublets at δ 4.58 and 4.67 ppm with a geminal coupling constant $^2J_{HH}$ of 14.10 Hz due to diastereotopic methylene protons on C^3. The 2-H proton resonated at δ 4.85 ppm and showed couplings with the methylene protons (^3J = 9.50, 2.60 Hz). The signal from the OH proton was located as δ 3.25 ppm (br.s). In the ^{1}H–^{13}C HMQC spectrum of **1** we observed cross-peaks between the methylene protons and C^3 (δ_C 74.14 ppm) and between 2-H and C^2 (δ_C 67.75 ppm). The CF_3 carbon signal was observed at δ_C 123.14 ppm. The ^{1}H–^{13}C HMBC spectrum of **1** (Fig. 1) showed correlations of 2-H with C^3 and C^1, and of each 3-H proton with C^2 and C^1.

The ^{1}H NMR spectrum of **2** corresponded to an *AB* spin system. The coupling constant $^3J_{AB}$ = 13.43 Hz indicated *E* configuration of the double C=C bond [20]. The H_B signal appeared downfield (δ 7.10 ppm) relative to H_A (δ 7.44 ppm) (Fig. 2). The given assignment of the H_A and H_B signals is confirmed by the corresponding ^{1}H–^{19}F coupling constants, $^3J(H_B–F)$ = 6.41, $^4J(H_A–F)$ = 1.22 Hz. The C^1 signal of **2** was located in a weaker field (δ_C 145.48 ppm, J_{CH} = 197.26 Hz) than that of C^2 (δ_C 125.74 ppm, $^2J_{CF}$ = 37.06 Hz), and the CF_3 carbon nucleus resonated at δ_C 121.12 ppm (J_{CF} = 270.93 Hz).

Independent evidences in favor of the assigned configuration of 3,3,3-trifluoro-1-nitropropene (**2**) were

obtained by DFT B3PW91/6-311++G(*df*,*p*) quantum chemical calculations of the relative energies of the *Z* and *E* isomers. The *E* isomer of **2** with the nitro group lying in the C=C bond plane was found to have the lowest energy. The energy of the *Z* isomer was higher by 26.1 kJ/mol [21]. In addition, the dipole moments of *Z*-**2** and *E*-**2** were calculated and measured experimentally.

Diastereoisomeric dibromide mixture **3a**/**3b** gave rise to double sets of signals in the ^{1}H, ^{13}C and ^{19}F NMR spectra (Fig. 3), the ratio **3a**/**3b** being 4:1 (**3a** is the stereoisomer characterized by lower chemical shifts). The IR spectrum of **3a**/**3b** contained absorption bands at 1581 and 1350 cm^{-1} due to stretching vibrations of the non-conjugated nitro group. For comparison, stretching vibration bands of the conjugated nitro groups in **2** and **4** were observed at 1555, 1307 and 1567, 1304 cm^{-1}, respectively.

Analysis of the ^{1}H and ^{13}C NMR spectra of compounds **4** and **2** and their analogs containing CCl_3 and CBr_3 groups [19, 21] allowed us to assign *Z* configuration to **4** (*trans* orientation of the trifluoromethyl and nitro groups). In the ^{1}H NMR spectrum of **4**, the 2-H proton resonated at δ 7.70 ppm with a coupling constant $^{3}J_{HF}$ of 6.46 Hz.

The ability of bromonitropropene **4** to act as reactive dipolarophile was demonstrated by the formation of 1,3-dipolar cycloaddition products in the reaction with phenyl azide. The reaction of **4** with phenyl azide at room temperature was complete in 14 days, and the product was a mixture of regioisomeric 1,2,3-triazoles **6a** and **6b** at a ratio of 5:1 (Scheme 3). Initially formed dihydrotriazoles **5a** and **5b** were detected in the reaction mixture by ^{1}H NMR (**5a**: $\delta_{4\text{-H}}$ 5.19 ppm; **5b**: $\delta_{5\text{-H}}$ 4.64 ppm); they lose HBr during chromatography on silica gel.

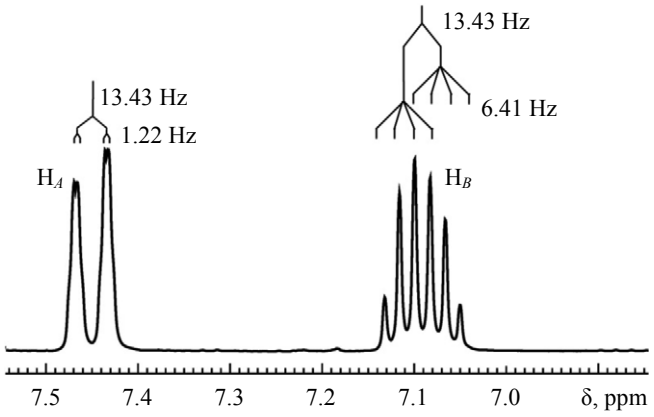


Fig. 2. ^{1}H NMR spectrum of 3,3,3-trifluoro-1-nitropropene (**2**) in $CDCl_3$.

The ^{1}H NMR spectrum of **6a**/**6b** contained only signals of aromatic protons in the region δ 7.18–7.68 ppm as three multiplets for each isomer. Protons of the benzene ring in **6a** resonated in a weaker field due to effect of the neighboring nitro group (δ 7.50, 7.62, 7.68 ppm); the corresponding signals of **6b** were observed at δ 7.18, 7.25, and 7.36 ppm. Major isomer **6a** was isolated in the pure state by chromatography, and its structure was determined by X-ray analysis.

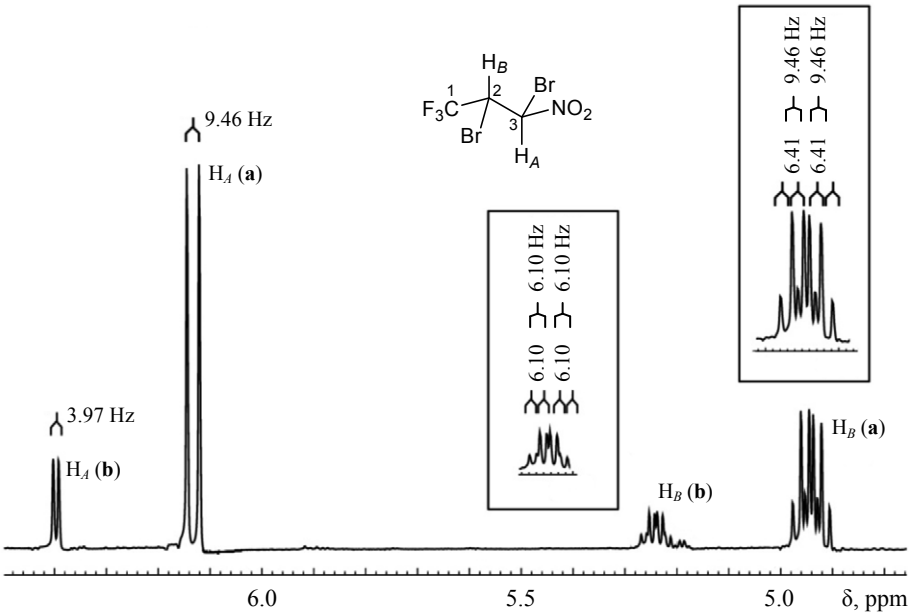
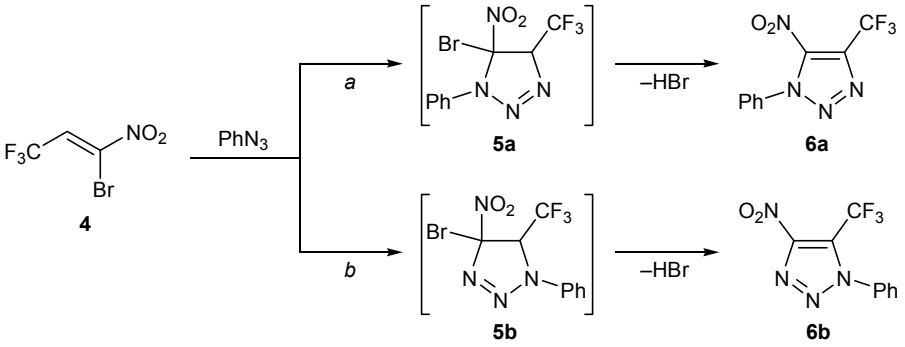


Fig. 3. ^{1}H NMR spectrum of diastereoisomer mixture **3a**/**3b**.

Scheme 3.



According to the X-ray diffraction data, the triazole ring in molecule **6a** is planar, and the nitro group lies almost in the same plane [the corresponding dihedral angle is 8.2(4)°]. The benzene ring is turned through a dihedral angle of 74.69(7)° with respect to the triazole ring plane (Fig. 4). The bond lengths and bond angles in molecule **6a** coincided within 3σ with the corresponding reference values [22].[1] The geometric parameters of the triazole ring in **6a** are similar to those of structural analogs found in the Cambridge Crystallographic Database: 1-(4-methoxyphenyl)-4-(trifluoromethyl)-1*H*-1,2,3-triazole (CCDC refcode KIBBUS) [23] and 4-(chloromethyl)-1-(4-methoxyphenyl)-5-(4-nitrophenyl)-1*H*-1,2,3-triazole (SUMWAZ) [24].

Packing of molecules **6a** in crystal is characterized by short intermolecular contacts $NO_2 \cdots H_{arom}$ ($O^2 \cdots H$ 62.63 Å) and $C^3F \cdots FC^3$ [$C^3 \cdots F^2$ 2.745(3) Å], as well as by $O \cdots \pi$ interaction between the O^2 atom and π-system of the benzene ring of the neighboring molecule [the distance between O^2 and the centroid of the benzene ring is 3.164(2) Å].

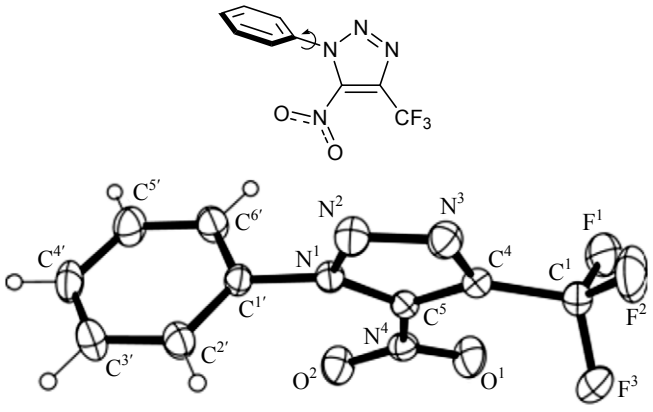


Fig. 4. Structure of the molecule of 4-nitro-5-(trifluoromethyl)-1-phenyl-1*H*-1,2,3-triazole (**6a**) according to the X-ray diffraction data.

In summary, we have developed a procedure for the synthesis of previously unknown 1-bromo-3,3,3-trifluoro-1-nitropropene and improved the procedure for the preparation of its precursor, 3,3,3-trifluoro-1-nitropropene. Using the reaction of 1-bromo-3,3,3-trifluoro-1-nitropropene with phenyl azide as an example, we have demonstrated the possibility of studying chemical transformations of this readily volatile and lachrymatory substance in ether solution without isolation in the pure state.

EXPERIMENTAL

The spectral studies were performed using the equipment of the Joint Center at the Faculty of Chemistry, Herzen State Pedagogical University of Russia. The ^1H, $^{13}C–\{^1H\}$, and $^1H–^{13}C$ HMBC NMR spectra were recorded on a Jeol ECX400A spectrometer (399.78 MHz for ^1H) using the residual proton and carbon signals of the deuterated solvent as reference. The IR spectra were measured on a Shimadzu IR Prestige-21 spectrometer from solutions in chloroform.

The products were isolated by column chromatography on Macherey-Nagel L silica gel (140/270 mesh) at a substrate-to-sorbent weight ratio of about 1:10 using eluotropic solvent series [25]. Silufol UV 254 plates were used for TLC; eluent hexane–acetone (2:1); spots were visualized by treatment with iodine vapor or under UV light. The ratios **3a**/**3b** and **5a**/**5b** were determined by ^1H NMR.

Trifluorocaetaldehyde hydrate was synthesized according to modified procedure [17].

The X-ray analysis of a single crystal of **6a** was performed at the X-Ray Analysis Laboratory, Joint Chemical Service Center, Vorozhtsov Novosibirsk Institute of Organic Chemistry, Siberian Branch,

[1] Tables containing geometric parameters of molecule **6a** are available from the authors by e-mail.

Russian Academy of Sciences. The data were acquired at room temperature on a Bruker Kappa APEX diffractometer (MoK_α radiation, graphite monochromator, CCD detector, $2\theta_{max}$ = 52.0°) from a 0.30×0.30×0.04-mm single crystal; $C_9H_5F_3N_4O_2$; monoclinic crystal system, space group *Pc*; unit cell parameters: *a* = 11.4132(6), *b* = 5.2817(2), *c* = 9.1242(5) Å; β = 111.517(2)°; *V* = 511.69(4) Å^3; *Z* = 2; d_{calc} = 1.676 g×cm^{-3}; μ = 0.158 mm^{-1}. Intensities of 1878 independent reflections were measured. A correction for absorption was applied using SADABS (transmission 0.68–0.75) [26]. The structure was solved by the direct method (SHELXS-97) [27] and was refined in anisotropic approximation (isotropic for hydrogen atoms) (SHELXL-97) [27]. The positions of hydrogen atoms were calculated on the basis of geometry considerations and were refined according to the riding model. Final divergence factors: wR_2 = 0.1049 for all 1878 independent reflections, *R* = 0.0436 for 1836 reflections with $F > 4\sigma(F)$; goodness of fit *S* = 1.13; 163 variables. The CIF file containing complete data set for structure **6a** was deposited to the Cambridge Crystallographic Data Centre (CCDC entry no. 1472942).

1,1,1-Trifluoro-3-nitropropan-2-ol (1). A mixture of 46.15 g (0.239 mol) of a 60% aqueous solution of trifluoroacetaldehyde hydrate, 14.58 g (0.239 mol, 12.8 mL) of nitromethane, and 1.99 g (0.019 mol) of sodium carbonate was vigorously stirred at 60°C until it became orange. The mixture was kept for 5 h at that temperature and treated with diethyl ether (2×50 mL). The combined extracts were dried over magnesium sulfate and evaporated on a rotary evaporator, and the residue was distilled at 85–86°C (18 mm) to obtain 35.72 g (94%) of **1** as an orange oil, R_f 0.70; published data [18]: bp 84°C (17 mm), yield 47%. IR spectrum, ν, cm^{-1}: 3603 (OH), 1568, 1384 (NO_2).

3,3,3-Trifluoro-1-nitropropene (2). Compound **1**, 35.72 g (0.225 mol) was distilled over P_2O_5 (36.35 g, 0.256 mol) under atmospheric pressure. Yield 24.11 g (76%), yellow–green liquid (strong lachrymator), bp 85–88°C; published data [17]: bp 89–90°C, yield 68%. IR spectrum ($CHCl_3$), ν, cm^{-1}: 1680 (C=C), 1555, 1307 (NO_2).

2,3-Dibromo-1,1,1-trifluoro-3-nitropropane (3a/3b). A mixture of 25.00 g (0.177 mol) of alkene **2** and 28.32 g (0.177 mol) of bromine was kept for 48 h at –20°C and poured into a Petri dish. Evaporation of unreacted bromine gave 43.16 g (81%) of a mixture of diastereoisomers **3a** and **3b** at a ratio of 4:1 (^{1}H NMR) as a highly volatile liquid. ^{1}H NMR spectrum, δ, ppm: **3a**: 6.15 (H_A), 4.95 (H_B); **3b**: 6.40 (H_A), 5.25 ppm (H_B). ^{13}C NMR spectrum, δ_C, ppm, **3a**: 121.70 q (C^1, $^1J_{CF}$ = 279.23 Hz), 44.28 q (C^2, $^2J_{CF}$ = 33.23, $^1J_{CH}$ = 162.45 Hz), 73.23 d (C^3, $^1J_{CH}$ = 180.59 Hz); **3b**: 126.00 q (C^1, $^1J_{CF}$ = 277.96 Hz), 47.20 q (C^2, $^2J_{CF}$ = 32.59 Hz), 78.89 d (C^3, $^1J_{CH}$ = 177.30 Hz). ^{19}F NMR spectrum, δ_F, ppm: –65.00 s (**3a**), –68.80 s (**3b**). Elemental analysis of **3a**/**3b** could not be obtained because of its high volatility.

1-Bromo-3,3,3-trifluoro-1-nitropropene (4). A solution of 13.34 g (0.143 mol, 14 mL) of 2-methylpyridine in 20 mL of diethyl ether was added dropwise with vigorous stirring at room temperature to a solution of 43.16 g (0.143 mol) of isomer mixture **3a**/**3b** in 50 mL of diethyl ether. The mixture was stirred for 40 min, ~20 mL of water was added, and the mixture was stirred for 30 min more. The organic layer was separated and dried over magnesium sulfate. The solvent was partially (~8–10 mL) removed on a rotary evaporator to obtain 32.17 g of a solution containing ~80% (^{1}H NMR) of **4** as a yellow–green volatile oil (strong lachrymator) which decomposed at room temperature (after complete removal of the solvent, the residue instantaneously warmed up and became charred), R_f 0.51. IR spectrum, ν, cm^{-1}: 1567, 1304 (NO_2); 1650 (C=C). UV spectrum (EtOH): λ 230 nm. ^{1}H NMR spectrum ($CDCl_3$): δ 7.70 ppm, q ($^3J_{HF}$ = 6.46 Hz). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 138.0 (C^1, $^3J_{CF}$ = 7.22 Hz), 125.53 (C^2, $^1J_{CH}$ = 174.04, $^2J_{CF}$ = 38.98 Hz), 120.63 (C^3, $^1J_{CF}$ = 271.57). ^{19}F NMR spectrum ($CDCl_3$): δ_F –61.0 ppm. All spectra were recorded from solutions containing a small amount of diethyl ether. Elemental analysis of **4** could not be obtained because of its high volatility and instability.

4-Nitro-5-(trifluoromethyl)-1-phenyl-1*H*-1,2,3-triazole (6a) and 5-nitro-4-(trifluoromethyl)-1-phenyl-1*H*-1,2,3-triazole (6b). Phenyl azide, 0.6 g (5 mmol), was added to a diethyl ether solution containing ~1.0 g (4.5 mmol) of compound **4**, and the mixture was kept for 14 days at 20°C. The precipitate, 0.37 g (32%) of a mixture of **6a** and **6b** at a ratio of 5:1, was filtered off. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 7.18 m, 7.29 m, 7.36 m, 7.50 m, 7.64 m, 7.68 m. According to the ^{1}H NMR data, the filtrate contained dihydrotriazoles **5a** and **5b** at a ratio of 3:2 [δ 5.19 m (1H, 4-H), 4.64 m (1H, 5-H)] and triazoles **6a** and **6b**. The filtrate was evaporated, and the residue was subjected to silica gel chromatography. Elution with hexane gave 0.51 g (44%) of a mixture of **6a** and **6b** at a ratio of 6:1. After repeated chromatography on silica

gel with hexane as eluent, from the second eluate portion (~100 mL) we isolated 0.29 g (25%) of **6a** with mp 93–95°C. IR spectrum, ν, cm^{-1}: 1547, 1386 (NO_2). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 7.50 m, 7.64 m, 7.68 m (C_6H_5). Found, %: C 41.82, 41.85; H 1.93, 1.97; N 21.67, 21.71. $C_9H_5F_3N_4O_2$. Calculated, %: C 41.86; H 1.94; N 21.71.

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