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LETTERS TO THE EDITOR

N,N'-Bis(trifluoromethanesulfonyl)oxamide

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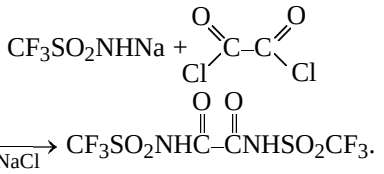
It is known that sulfonamides RSO_2NH_2 react with oxalyl chloride both by way of substitution to give bis(sulfonyl)oxamides $RSO_2NHC(O)C(O)NHSO_2R$ and in a more complicated fashion, with decarbonylation and cyclization, to form sulfonyl isocyanates RSO_2NCO and parabanic acid derivatives [1]. The same reaction with trifluoromethanesulfonamide $CF_3SO_2NH_2$ would be expected to give the oxamide $CF_3SO_2NHCOCONHSO_2CF_3$ (**I**) which is a strong NH acid. Lithium salts of such acids are used as ionogenic additives to nonaqueous electrolytes in chemical sources of current [2, 3]. We found that compound **I** is formed by the reaction of sodium trifluoromethanesufonamide and oxalyl chloride. The structure of the reaction product was confirmed by its ^1H, ^{13}C, and ^{19}F NMR and IR spectra and elemental analysis.



$$CF_3SO_2NHNa + \underset{Cl}{\overset{O}{\diagdown}}C\text{–}C\underset{Cl}{\overset{O}{\diagup}} \xrightarrow[-NaCl]{} CF_3SO_2NHC(O)\text{–}C(O)NHSO_2CF_3.$$

N,N'-Bis(trifluoromethanesulfonyl)oxamide easily hydrolyses. Its ^{13}C NMR spectrum in D_2O contains a quartet at 118.28 ppm ($^1J_{CF}$ 320.13 Hz) due to the CF_3 group of the starting amide and shows no carbonyl carbon signal and the quartet of the CF_3 group of *N,N'*-bis(trifluoromethanesulfonyl)oxamide.

$$CF_3SO_2NHC(O)\text{–}C(O)NHSO_2CF_3 \xrightarrow[-CO,\ -CO_2]{H_2O} CF_3SO_2NH_2.$$

***N,N'*-Bis(trifluoromethanesulfonyl)oxamide.** Trifluoromethanesulfonamide, 7.45 g, was treated with the solution of sodium methoxide, prepared from 1.13 g of sodium in 50 ml of methanol. The resulting solution was evaporated to dry, and the residue was dried in a vacuum. Tetrahydrofuran, 25 ml, was added to the dried salt CF_3SO_2NHNa and then, dropwise with stirring, 3 ml of oxalyl chloride. The mixture was stirred for 1 h and evaporated to dry. The residue was treated with anhydrous ether, stirred, the sodium chloride was filtered off, the solvent was removed, and the residue was purified by vacuum sublimation at 130°C. mp 170–175°C. IR spectrum (KBr), ν, cm^{-1}: 3200 (NH), 1750 (CO), 1370 and 1120 (SO_2), 1200 (C–F); 1430 (NH). ^1H NMR spectrum (THF-d_8), δ, ppm: 10.33 s (NH). ^{13}C NMR spectrum (THF-d_8), δ_C, ppm: 118.60 q (CF_3, $^1J_{CF}$ 321.8 Hz), 155.58 (CO). ^{19}F NMR spectrum (THF-d_8), δ_F, ppm: –71.67. Found, %: C 13.15; H 0.87; F 18.09; N 8.43; S 18.09. $C_4H_2F_6N_2O_6S_2$. Calculated, %: C 13.64; H 0.57; F 32.37; N 7.95; S 18.21.

The IR spectra were recorded on a Specord IR-75 spectrometer. The NMR spectra were obtained on a Bruker DPX-400 spectrometer, working frequences 400 (^1H), 100 (^{13}C), and 376 MHz (^{19}F). The chemical shifts were measured against HMDS (^1H, ^{13}C) and $CFCl_3$ (^{19}F).

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