cold water and extracted with hexane, and the organic layer was washed with water and dried with $CaCl_2$. The substance obtained after evaporation of the solvent (2.54 g) was analyzed by TLC and ^{31}P NMR spectroscopy. The mixture was found to contain the starting compound (Ph_3PO, δ 24.94) and triphenylphosphine (PPh_3, δ −6.15). The current yield of the latter was 24 % and the substance yield was 30.5 %, judging from the integral intensities of the corresponding signals in the ^{31}P NMR spectrum. Aluminum is ionized at the anode (the current yield of Al^{3+} is 140 %), while at the cathode, the Al^{3+} ions are reduced. Apparently, the process of the formation of Ph_3P includes the generation of aluminum ions in a low oxidation state or Al^0, which then reduce triphenylphosphine oxide.

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Synthesis of trifluoroacetates of *N*-(2-hydroxyethyl)-substituted trifluoroacetaminosuccinodiamide and succinimide

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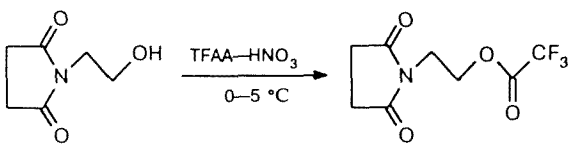
Previously[1] it was demonstrated that the treatment of mannitol and sorbitol with a trifluoroacetic anhydride—nitric acid system (TFAA—HNO_3) resulted in the formation of the corresponding hexanitrates in 45 % yields. Similar results were obtained on the nitration of 1,1,4,4-tetranitrobutan-2,3-diol and its derivatives.[2]

We have found that another reaction pathway takes place when *N*-(2-hydroxyethyl)succinimide is treated with the same system, namely, acylation of the hydroxy group occurred instead of *O*-nitration (Scheme 1).

Scheme 1



1

Trifluoroacetate **1** was isolated as a white crystalline substance in a 43 % yield when the reaction mixture was diluted with water; m.p. 87—89 °C. ^1H NMR (CD_3CN, $SiMe_4$), δ: 2.60 (s, 4 H, CH_2); 3.78 (t, 2 H, NCH_2, $^3J_{CH_2-CH_2}$ = 6.0 Hz); 4.44 (t, 2 H, $CH_2OC(O)$, $^3J_{CH_2-CH_2}$ = 6.0 Hz). IR ($ClCH_2CH_2Cl$, film), ν/cm^{-1}: 1111, 1159 (C—F); 1225 (C(O)O); 1694 (C=O, imide); 1796 (C=O in $CF_3C(O)O$); 2922, 2951, 2982 (CH_2).

In the case of *N*,*N*'-bis(2-hydroxyethyl)trifluoroacetaminosuccinodiamide, which contains N—H bonds in amide groups, *N*-nitration of the amide nitrogen atoms occurs along with *O*-acylation (Scheme 2).

Compound **2**, which was hitherto unknown, was obtained as a white crystalline substance in a 95 % yield; m.p. 79.0—80.5 °C (from a CCl_4—$ClCH_2CH_2Cl$ mixture). ^1H NMR (CD_2Cl_2, $SiMe_4$), δ: 3.73 (m, 2 H, CH_2O, ABX-system, Δν = 31.6 Hz, $|^2J_{AB}|$ = 18.3 Hz, $^3J_{AX}$ = 5.1 Hz, $^3J_{BX}$ = 5.5 Hz); 4.40—4.70 (m, 8 H, OCH_2CH_2N); 5.90 (br.m, 1 H, CH, ABX-system); 7.38 (br.d, 1 H, $NHCO(CF_3)$, $^3J_{NH-CH}$ = 7.5 Hz). IR (KBr),

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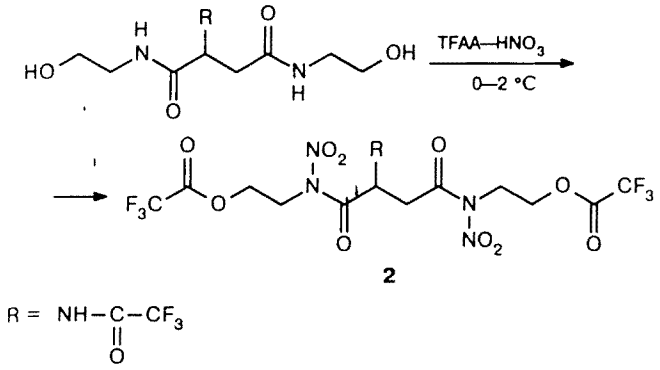
Scheme 2



ν/cm^{-1}: 1178, 1199 (C—F); 1235 (C—O); 1292, 1601 (N—NO_2); 1709 (C=O, amide); 1793 (C=O in $CF_3C(O)O$); 3308 (NH).

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New one-pot synthesis of α-hydroxyaldehyde ethyl sulfates from terminal olefins

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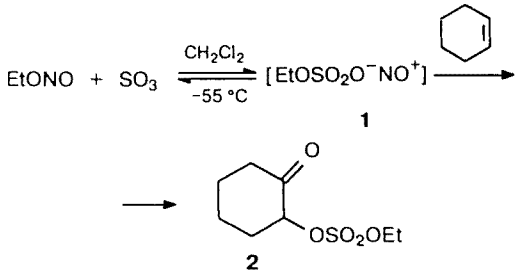
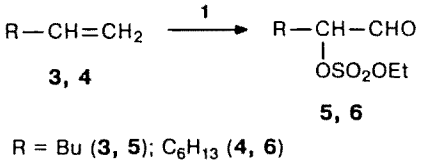
The search for new electrophilic reagents or ways to activate known substrates to increase their electrophilicity are of great importance for synthetic organic chemistry. The method based on the insertion of sulfur trioxide into weak electrophiles or compounds exhibiting no electrophilic properties is an example of such activation.[1,2] Previously, we have described the reaction of SO_3 with ethyl nitrite (EtONO) affording nitrosonium ethyl sulfate (**1**). This compound is stable in methylene chloride solution up to −30 °C and reacts easily with various cyclic olefins[3] to give ketosulfates **2**.



We studied the reaction of nitrosonium ethyl sulfate with terminal olefins. Under these conditions, hex-1-ene (**3**) and oct-1-ene (**4**) give the corresponding α-hydroxyaldehyde ethyl sulfates **5** and **6** in 70 % yields.



It should be noted that these yields are given for individual compounds isolated by column chromatography. Thus, nitrosonium ethyl sulfate is a convenient synthetic reagent for the transformation of terminal olefins to the derivatives of hydroxyaldehydes. Certainly, its synthetic abilities are not restricted to the reactions described above, and their studies are now in progress.

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