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# Reaction of *N*-cyanoamines with polyfluorocarbonyl compounds

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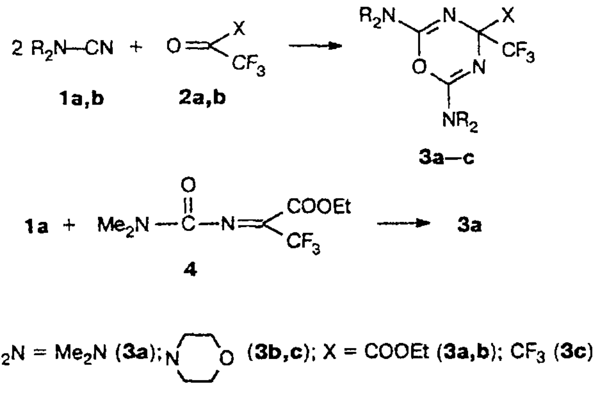
In a study of reactions of *N*-cyanodimethylamine (**1a**) and *N*-cyanomorpholine (**1b**) with ethyl 3,3,3-trifluoropyruvate (**2a**) and hexafluoroacetone (**2b**), we discovered a new type of transformation of cyanoamines, which give 1,3,5-oxadiazines (**3a—c**) with polyfluorocarbonyl compounds.



The structures of the compounds obtained were proved by $^1$H and $^{19}$F NMR spectroscopy and by data of elemental analysis; the structure of **3a** was confirmed by its alternative synthesis from **1a** and ethyl 2-*N*-(*N*,*N*-dimethylcarbamoyl)imino-3,3,3-trifluoropropionate (**4**) using the previously described procedure.[1]

**Ethyl 2,6-bis(dimethylamino)-4-trifluoromethyl-1,3,5-oxadiazine-4-carboxylate (3a).** *A.* A mixture of cyanide **1a** (0.01 mol) and ketone **2a** (0.05 mol) in 50 mL of anhydrous ether was kept for 24 h at ~20 °C. The ether was evaporated, and the residue was recrystallized from a hexane—benzene mixture (10 : 1) to give oxadiazine **3a**, yield 89%, m.p. 90—92 °C. Found (%): C, 42.69; H, 5.28; N, 17.83. $C_{11}H_{17}F_3N_4O_3$. Calculated (%): C, 42.60; H, 5.53; N, 18.07. $^1$H NMR ($CDCl_3$), δ: 1.28 (t, 3 H, $\underline{Me}CH_2$); 2.95 (s, 12 H, MeN); 4.22 (q, 2 H, $CH_2O$). $^{19}$F NMR ($CDCl_3$), δ: −3.11.

***B.*** A mixture of compound **1a** (0.01 mol) and acylimine **4** (0.01 mol) in 50 mL of anhydrous ether was kept for 24 h at ~20 °C. The ether was evaporated, and the residue was recrystallized from a hexane—benzene mixture (10 : 1) to give oxadiazine **3a**, yield 92%, m.p. 90—92 °C. The NMR characteristics of the product were identical with those of the sample of **3a** prepared by procedure *A*.

**Ethyl 2,6-bis(morpholino)-4-trifluoromethyl-1,3,5-oxadiazine-4-carboxylate (3b)** was prepared similarly to compound **3a** (procedure *A*). Yield 87%, m.p. 91—92 °C. Found (%): C, 45.90; H, 5.38; N, 14.63. $C_{15}H_{21}F_3N_4O_5$. Calculated (%): C, 45.71; H, 5.37; N, 14.21. $^1$H NMR ($CDCl_3$), δ: 1.30 (t, 3 H, $\underline{Me}CH_2$); 3.44 (m, 4 H, $CH_2N$); 3.70 (m, 4 H, $CH_2O$); 4.24 (q, 2 H, $CH_2O$). $^{19}$F NMR ($CDCl_3$), δ: −2.95.

**2,6-Bis(morpholino)-4,4-bis(trifluoromethyl)-1,3,5-oxadiazine (3c).** A mixture of cyanide **1b** (0.01 mol) and ketone **2b** (0.05 mol) in 50 mL of anhydrous ether was kept in a closed vessel for 24 h at ~20 °C. The ether was evaporated, and

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the residue was recrystallized from a hexane—benzene mixture (10 : 1) to give oxadiazine **3c**, yield 93%, m.p. 193—195 °C. Found (%): C, 39.89; H, 4.27; N, 14.43. $C_{13}H_{16}F_6N_4O_3$. Calculated (%): C, 40.02; H, 4.13; N, 14.36. $^1H$ NMR ($CDCl_3$), δ: 3.44 (m, 4 H, $CH_2N$), 3.69 (m, 4 H, $CH_2O$). $^{19}F$ NMR ($CDCl_3$), δ: −3.04.

The $^1H$ and $^{19}F$ NMR spectra were recorded on a Bruker CXP 200 spectrometer. Melting points were measured in a capillary.

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# 2,2'-Ethylenedi-1-boraadamantane — a new derivative of 1-boraadamantane

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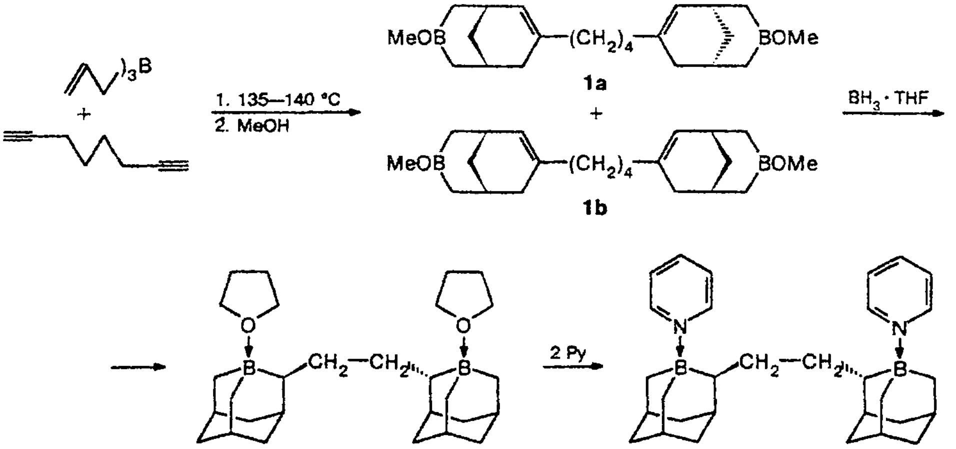
1-Boraadamantane and its homologs possess enhanced chemical activity due to the tetrahedral ($sp^3$) configuration of the boron atom,[1] which is unusual for triorganoboranes $R_3B$. Based on these compounds, we developed preparative methods for the synthesis of different types of cyclic, bicyclic, and cage compounds, including 1,3,5-trimethylenecyclohexane,[2] remantadine,[3] and 1-azaadamantanes.[4] In this work, we report on the first synthesis of a compound with two 1-boraadamantane fragments in the molecule.

The reaction of triallylborane with octa-1,7-diyne at 135—140 °C (allylboron—acetylene condensation[5]) results (after treatment with methanol) in the formation of 7,7'-tetramethylenedi(3-methoxy-3-borabicyclo[3.3.1]non-6-ene) (**1**), probably, as a mixture of diastereomers **1a** and **1b**.

Hydroboration of **1** by treatment with a solution of $BH_3 \cdot THF$ in THF according to the known procedure[6] followed by heating at 66 °C for 2 h afforded a tetrahydrofuran complex of 2,2'-ethylenedi-1-boraadamantane



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