trum); 4.72 (2 H, $CF(NO_2)CH_2$, AB is the portion of the ABX spectrum); 4.75 (s, 2 H, OCH_2O). IR, ν/cm^{-1}; 653 (NO_2); 800, 851 (C—N); 1052 (C—F and C—O); 1130 (C—O); 1223 (C—O) from —C(O)—O); 1316, 1580 (C—NO_2 from $FC(NO_2)$); 1607 (C—NO_2) from $FC(NO_2)_2$—); 1760 (C=O); 1415, 1454, 2915, 2960 (CH_2).

Under identical conditions, ether **5** (colorless oil, n_D^{20} 1.4508) forms from **2** and NH_2OH in a yield of ~48 %. Found (%): C, 21.64; H, 2.50; F, 13.76; N, 15.09. $C_5H_7F_2N_3O_8$. Calculated (%): C, 21.82; H, 2.55; F, 13.82; N, 15.27. ^1H NMR (CD_3CN), δ: 4.05 (2 H, $CF(NO_2)CH_2OH$, AB is the portion of the ABX spectrum); 4.30 (2 H, $OCH_2CF(NO_2)$, AB is the portion of the ABX spectrum); 4.65 (br.s, H, OH); 4.85 (CH_2, $CF(NO_2)CH_2O$, AB is the portion of the ABX spectrum). IR, ν/cm^{-1}; 764 (NO_2); 800, 851 (C—N); 1067 (C—OH overlaps with C—F); 1148 (C—O); 1313, 1547 (NO_2 from $CF(NO_2)$); 1604 (NO_2) from $FC(NO_2)_2$); 2925, 2946 (CH_2), 3416, 3579 (OH).

The corresponding acetate (colorless oil, n_D^{20} 1.4420, purity 99.5 % (GLC)) was obtained upon treatment of ether **5** with acetyl chloride in a yield of 93 %. ^1H NMR (CD_3CN), δ: 2.03 (s, 3 H, CH_3); 4.4 (2 H, $OCH_2CF(NO_2)$, AB is the portion of the ABX spectrum); 4.65 (2 H, $CF(NO_2)CH_2OCO$, AB is the portion of the ABX spectrum); 4.85 (2 H, $CF(NO_2)_2CH_2$, AB is the ABX spectrum). All spectra of the ABX type exhibit strong interactions, Δν, and *J* were not determined.

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Preparative synthesis of dimethylaluminum hydride

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Methylaluminoxanes (MAO) are widely used for polymerization of olefins.[1,2] The synthesis of MAO by the incomplete hydrolysis of Me_3Al is complicated by the formation of $Al(OH)_3$, instability of the composition and the structure of MAO, *etc.*[3] It is conceivable that the substitution of Me_2AlH for Me_3Al could result in controlled hydrolysis because of the insignificant difference in bond activity of Al—C and Al—H.

Since the known procedures for the synthesis of Me_2AlH make it difficult to isolate the target product in preparative amounts, we elaborated a simple method for the synthesis of Me_2AlH from Me_3Al and crystalline AlH_3, following the reaction:

$$2Me_3Al + AlH_3 \longrightarrow 3Me_2AlH.$$

The synthesis of nonsolvated AlH_3 free from organic admixtures was carried out through the crystallization of AlH_3 from an ether—toluene solution followed by the elimination of ether according the known procedure.[5]

The synthesis of Me_2AlH from crystalline AlH_3. A suspension of AlH_3 (6.5 g, 0.217 mol) in $AlMe_3$ (28.6 g, 0.397 mol) was stirred at 120—126 °C (Ar) for 0.5 h. Distillation from the same flask (air condenser, distillation "pig", receiving vessels) yielded Me_2AlH (26.5 g, 74 %), b.p. ~90 °C (70—90 Torr). (Me_2AlH becomes glass-like at ~20 °C, taking on sufficient mobility at ~90 °C, which determined the distillation conditions). During the distillation, Me_2AlH decomposed partially to metallic Al. A condensate solidified on the walls of the condenser and distillation "pig" and did not get to the receiving vessels. The distillation at atmospheric pressure is accompanied by considerable decomposition. Me_2AlH was obtained in a <60 % yield, b.p. 152—154 °C (*cf.* Ref. 4).

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Allyldeboration of organoboron compounds in aqueous media catalyzed by "ligandless" palladium

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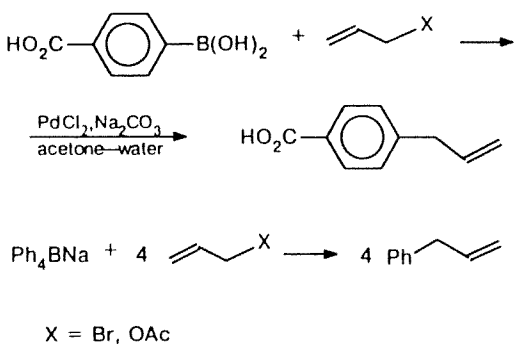
Reactions of allyl electrophilic agents with organoboron compounds, which are catalyzed by palladium or nickel complexes, are an important method of forming a new carbon—carbon bond.[1–4] They are usually carried out in an organic solvent with long heating.

We found that allyldeboration of organoboron compounds in aqueous acetone proceeded under very mild conditions at a high rate if "ligandless" palladium[5] was used as a catalyst. $PdCl_2$ was the source of this catalyst. The reaction of allyl bromide with 4-carboxyphenylboronic acid and sodium tetraphenylborate proceeds at room temperature for 3—6 h, resulting in high yields of 4-allylbenzoic acid (80 %) and allylbenzene (75 %). When less active allylacetate is used, the reaction proceeds at 56 °C for 2—3 h to give the products of allyldeboration in quantitative yields.



It should be noted that all four phenyl groups in Ph_4BNa participate in the reaction with both allylbromide and allyl acetate.

A 0.1 *M* aqueous solution of $PdCl_2$ (0.1 mL, 0.01 mmol) was added under argon to a mixture of a 1.7 *M* aqueous solution of Na_2CO_3 (1.77 mL, 3 mmol), acetone (5.3 mL), allylbromide (0.087 mL, 0.121 g, 1 mmol), and 4-carboxyphenylboronic acid (0.166 g, 1 mmol). The mixture obtained was stirred at ~20 °C for 6 h, and then diluted with water (75 mL) and filtered. The filtrate was cooled to 0 °C and acidified with HCl. The precipitate that formed was filtered off, washed with water, and dried *in vacuo* over P_2O_5. 4-Allylbenzoic acid (0.1255 g, 80 %) was obtained, m.p. 104—105 °C (*cf.* Ref. 6: 104—105 °C). ^{1}H NMR (400 MHz, $(CD_3)_2CO$), δ: 3.46 (d, 2 H, J = 6.4 Hz); 5.02—5.14 (m, 2 H); 5.92—6.4 (m, 1 H); 7.33 (d, 2 H, J = 8.0 Hz); 7.97 (d, 2 H, J = 8.0 Hz).

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