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Allylboration of some ketones and aldehydes with 2-allyl-1,2-oxaborolane. Isolation of their intermediate adducts and synthesis of homoallylic alcohols

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Abstract

2-Allyl-1,2-oxaborolane (**II**), prepared by the reaction of 2-allyloxy-1,2-oxaborolane (**I**) with allylmagnesium bromide in ether, is an extremely reactive allylborane. This cyclic borinate ester **II** is a BCCO-type organoborane and can be used as a novel allylboration reagent. As is usual with allylboranes, **II** can also add smoothly to various ketones or aldehydes, and when followed by deboronation with diethanolamine gives the corresponding homoallylic alcohols **IV**. The adducts **III**, formed *via* a six-centre cyclic mechanism and allylic rearrangement, have been isolated and identified as a kind of 2-alkenoxy-1,2-oxaborolane.

1. Introduction

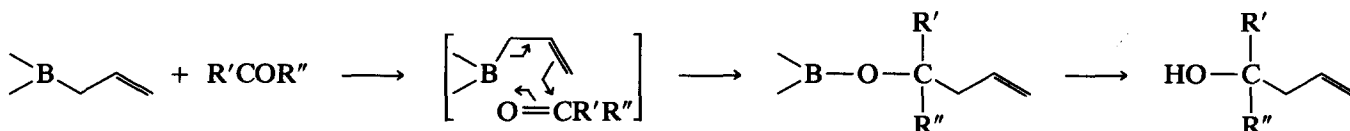
Mikhailov and Bubnov were the first to report that simple BCCC-type allylboranes, such as triallylborane [1], diallyl(alkyl)boranes and allyl(dialkyl)boranes, represent a class of highly reactive intermediates in organoboron chemistry [2,3]. In particular, such allylboranes can react with aldehydes and ketones, subsequent addition of the B-allyl fragment to the carbonyl group proceeding through allylic rearrangement [2–5]. Over two decades the allylboration of carbonyl compounds has been studied widely, however, up to now only a few organoborane adducts have been isolated as intermediates. A variety of allyl(alkyl) boranes (*e.g.* B-allyl-9-BBN *etc.*) have been utilized for the synthesis of homoallylic alcohols [2,6] (Scheme 1). Recently stud-

ies on many chiral allyl(dialkyl)boranes (allyl-BCC*) or allylboronate esters (allyl-BOO*) have made possible a fruitful approach to asymmetric synthesis [7].

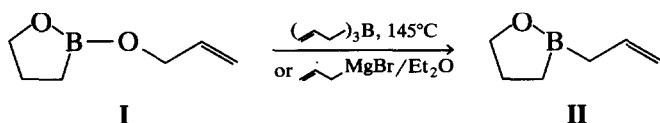
In 1982 we reported that 2-allyl-1,2-oxaborolane (**II**, allyl-BCO) is another novel allylborane [8,9] which remains stable in an inert atmosphere for a long period. The cyclic borinate ester **II** is easily prepared by the heat exchange reaction of triallylborane with 2-allyloxy-1,2-oxaborolane (**I**) or by Grignard reaction of **I** with allylmagnesium bromide in ethyl ether [8–10].

We have studied the physical and chemical properties of **II** in detail. B-Allylic rearrangement reactions of **II** include protolysis with water, alcohols or amines and allylboration of carbonyl compounds *via* C–C bond formation. A number of 2-alkoxy- and 2-amino-1,2-oxaborolanes has been synthesized in the former fashion, rupturing the B-allyl bond with the evolution of propene [10,11] (Scheme 3). In this paper we would like to report allylboration of 2-allyl-1,2-oxaborolane (**II**) with some aldehydes and ketones.

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Scheme 1.



Scheme 2.

2. Results and discussion

Allylboration of some ketones and aldehydes with 2-allyl-1,2-oxaborolane (**II**) proceeds smoothly without any solvent (Scheme 4). The conditions of reaction with aldehydes are milder (0°C–r.t.) than with ketones ($\geq 100^\circ\text{C}$). These adducts **III** can be isolated in good yields and have been identified by elemental analyses and IR, proton NMR and mass spectroscopies. The physical data of 2-alkenoxy-1,2-oxaborolanes **III** are listed in Table 1.

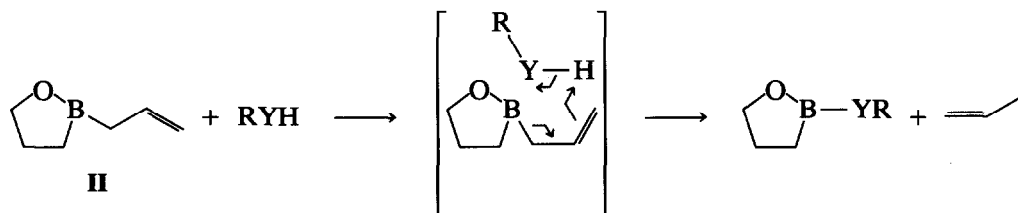
As with allyl(dialkyl)boranes (allyl–BCC), allylboration of α,β -unsaturated aldehydes, *e.g.* crotonaldehyde, with **II** (allyl–BCO) proceeds *via* 1,2-addition on the carbonyl group in order to satisfy the requirement of a six-membered transition state [14]. Only the 1,2-addition product **IIIh** was obtained. Allylborane **II** also

TABLE 1. Physical data for adducts **IIIa–j**

Formula III	Yield (%)	B.p. (°C/mbar)	n_D^{20}	d_4^{20}	MR (calcd. [13])
a $\text{BC}_9\text{H}_{17}\text{O}_2$	96	92–93/65	1.4381	0.9064	48.68 (48.61)
b $\text{BC}_{10}\text{H}_{19}\text{O}_2$	86	96–98/45	1.4447	0.9108	53.17 (53.26)
c $\text{BC}_{11}\text{H}_{21}\text{O}_2$	87	102–104/30	1.4459	0.9075	57.61 (57.91)
d $\text{BC}_{14}\text{H}_{19}\text{O}_2$	90	115–116/4.6	1.5132	1.0178	67.98 (68.28)
e $\text{BC}_{11}\text{H}_{19}\text{O}_2$	80	76–77/4.6	1.4681	0.9751	55.34 (55.67)
f $\text{BC}_{12}\text{H}_{21}\text{O}_2$	65	87–88/4	1.4721	0.9712	60.01 (60.50)
g $\text{BC}_9\text{H}_{17}\text{O}_2$	85	103–104/65	1.4473	0.9237	48.60 (48.61)
h $\text{BC}_{10}\text{H}_{17}\text{O}_2$	87	85–86/10	1.4633	0.9360	53.00 (52.78)
i $\text{BC}_{13}\text{H}_{17}\text{O}_2$	81	108–109/12	1.5173	1.0273	63.67 (63.69)
j $\text{BC}_9\text{H}_{14}\text{O}_2\text{F}_3$	70	62–63/20	1.4030	1.1180	48.46 (47.87)

reacts with fluoro-containing ketones, *e.g.* trifluoroacetone, to give the corresponding fluoro-containing derivatives. Spectroscopic data and elemental analyses of **III** confirmed the structures of 2-alkenoxy-1,2-oxaborolanes.

The intermediates **IIIa–j** can be hydrolyzed to yield the corresponding homoallyl alcohols **IVa–j** by re-esterification with diethanolamine. The products **IVa–j** are three-carbon atom enhanced β,γ -unsaturated carbinols. The alkenols **IV** can be converted into the corresponding **III** by treatment with **II**.



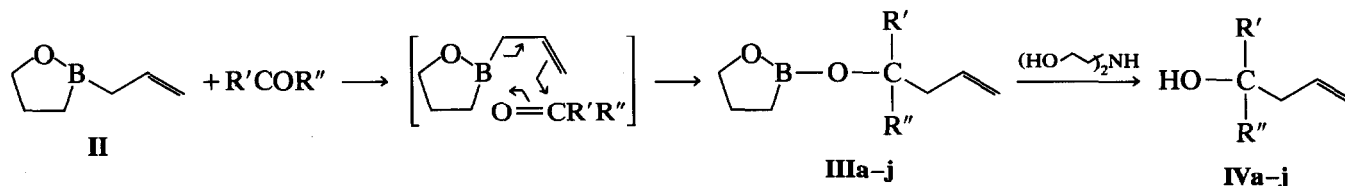
Y = O, R = Et [11];

Y = NH, R = Allyl, Pr, Bu, ^tBu, ^cC₆H₁₁, NMe₂;

Y = NR, R = Allyl, ⁱPr, Bu, ^cC₆H₁₁;

or R₂ = $-(\text{CH}_2)_5-$, $-\text{CH}_2\text{CH}_2\text{OCH}_2\text{CH}_2-$ [10].

Scheme 3.



a: R' = Me, R'' = Me;

b: R' = Me, R'' = Et;

c: R' = Me, R'' = Pr;

d: R' = Me, R'' = Ph;

e: R', R'' = $-(\text{CH}_2)_5-$;

f: R', R'' = $-(\text{CH}_2)_6-$;

g: R' = H, R'' = Et;

h: R' = H, R'' = $-\text{CH}=\text{CHCH}_3$;

i: R' = H, R'' = Ph;

j: R' = Me, R'' = CF₃.

Scheme 4.

3. Experimental details

All operations with the organoboranes were carried out under purified nitrogen. We prepared 2-allyloxy-1,2-oxaborolane (**I**) by dehydrogenation-hydroboration of allyl alcohol with $\text{KBH}_4\text{-HOAc}$ [10,12]. 2-Allyl-1,2-oxaborolane (**II**) was prepared in about 50% yield from **I** by the Grignard reaction [10]. IR spectra were recorded on a Specord 75 spectrophotometer. ^1H or ^{19}F NMR spectra were recorded on a Varian Model EM-360 (60 MHz) instrument relative to external TMS or trifluoroacetic acid respectively. ^{13}C NMR spectrum of **IIId** was recorded on an FX-90Q instrument. Mass spectra were determined with a Finnigan MS-4021 instrument. All reagents were purified before use.

Thermometers used were uncorrected.

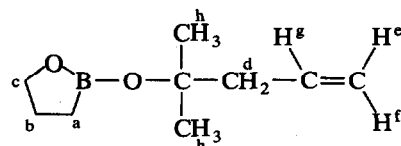
3.1. General procedure for allylboration of ketones with **II**

3.1.1. 2-(2-Methyl-4-penten-2-yloxy)-1,2-oxaborolane (**IIIa**)

A 25 ml dry, nitrogen-filled round bottomed flask equipped with a side arm capped with a rubber septum, a magnetic bar, a reflux condenser and a connecting tube attached to a bubbler, was charged with 7.70 g (70 mmol) of **II**. 4.07 g (70 mmol) of acetone was added dropwise during 10 min and then the reaction mixture was heated at 80–100°C for 3 h. Distillation under vacuum gave 11.3 g (96.1%) of **IIIa**, b.p. 97–98°C/80 mbar, n_D^{20} 1.4381.

Redistillation provided the analytical sample for determination of physical data (Table 1), spectroscopic and microanalytical data.

^1H NMR (CCl_4): δ 0.66 (t, 2H, $J = 7.8$, a); 1.10 (s, 6H, h); 1.66 (q, 2H, $J = 7.2$, b); 2.16 (d, 2H, $J = 3.5$, d); 3.79 (t, 2H, $J = 6.4$, c); 4.66, 4.86, 5.58 (m, 3H, e,f,g). IR (film): ν (cm^{-1}) 595w, 620w, 645w, 730w, 795w, 825w, 860w, 900s, 915s, 970m, 990s, 1030s, 1150s, 1170s, 1195s, 1225s, 1250s, 1275m, 1310m, 1360s, 1400s, 1445s, 1495s, 1640m, 2885s, 2980s, 3075m. MS (DEI): m/z (%) $M + 1$, 169 (13.3); M^+ , 168 (4.1); M -allyl, 127 (11.0); $M + 1 - \text{BC}_3\text{H}_6\text{O}_2$, 84 (100.0).

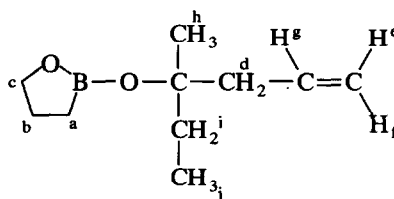


Anal. Found: B, 6.18; C, 64.00; H, 10.64. $\text{BC}_9\text{H}_{17}\text{O}_2$ calcd.: B, 6.43; C, 64.33; H, 10.20%.

3.1.2. 2-(3-Methyl-5-hexen-3-yloxy)-1,2-oxaborolane (**IIIb**)

As described above, the mixture of 7.70 g of **II** and 5.05 g (70 mmol) of 2-butanone was heated at 120°C for 3 h. 11.0 g (86.3%) of **IIIb** was obtained. B.p. 100–101°C/45 mbar, n_D^{20} 1.4445.

^1H NMR (CCl_4): δ 0.66 (t, 5H, $J = 6.8$, a,j); 1.05 (s, 3H, h); 1.52, 1.65 (q, 4H, $J = 7.0$, i,b); 2.20 (d, 2H, $J = 6.4$, d); 3.78 (t, 2H, $J = 6.5$, c); 4.68, 4.90, 5.58 (m, 3H, e,f,g). IR (film): ν (cm^{-1}) 910m, 990m, 1030m, 1125m, 1195s, 1250m, 1310m, 1360s, 1405s, 1415s, 1460s, 1490m, 1640m, 2880m, 2970s, 3075w. MS (DEI): m/z (%) $M + 1$, 183 (10.9); M^+ , 182 (3.1); M -allyl, 141 (21.3); $M - \text{BC}_3\text{H}_6\text{O}_2$, 99 (100.0).

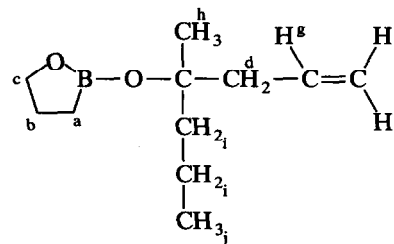


Anal. Found: B, 5.74; C, 65.86; H, 10.91. $\text{BC}_{10}\text{H}_{19}\text{O}_2$ calcd.: B, 5.94; C, 65.97; H, 10.52%.

3.1.3. 2-(4-Methyl-1-hepten-4-yloxy)-1,2-oxaborolane (**IIIc**)

The mixture of 7.70 g of **II** and 6.03 g (70 mmol) of 2-pentanone was heated at 140°C for 3 h. 11.8 g (86.0%) of **IIIc** was obtained. B.p. 106–107°C/35 mbar, n_D^{20} 1.4457.

^1H NMR (CCl_4): δ 0.64, 0.76 (m, 5H, a,j); 1.11 (s, 3H, h); 1.33, 1.64 (m, 6H, i,b); 2.20 (d, 2H, $J = 6.4$, d); 3.80 (t, 2H, $J = 6.8$, c); 4.73, 4.93, 5.63 (m, 3H, e,f,g). IR (film): ν (cm^{-1}) 740w, 795w, 825w, 855w, 880w, 915s, 950m, 970m, 990s, 1025s, 1150m, 1170m, 1195s, 1250s, 1310m, 1360s, 1400s, 1450s, 1490s, 1640m, 2875s, 2960s, 3070m.

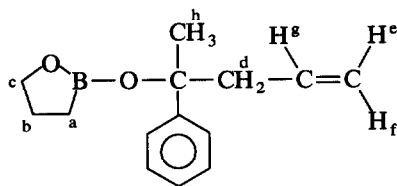


Anal. Found: B, 5.38; C, 67.00; H, 11.24. $\text{BC}_{11}\text{H}_{21}\text{O}_2$ calcd.: B, 5.51; C, 67.37; H, 10.79%.

3.1.4. 2-(2-Phenyl-4-penten-2-yloxy)-1,2-oxaborolane (**IIId**)

As above, starting from 7.70 g of **II** and 8.41 g (70 mmol) of acetophenone, 20.7 g (90.0%) of **IIId** was prepared. B.p. 115–116°C/4.6 mbar, n_D^{20} 1.5132.

^1H NMR (CCl_4): δ 0.73 (t, 2H, $J = 8$, a); 1.63 (s, 3H, h); 1.73 (q, 2H, $J = 7.5$, b); 2.77 (d, 2H, $J = 6.2$, d); 3.87 (t, 2H, $J = 7$, c); 4.7–6.1 (m, 3H, e,f,g); 7.0–7.6 (m, 5H, Ph). IR (film): ν (cm^{-1}) 665s, 700s, 760m, 910m, 990m, 1015s, 1070w, 1160w, 1200m, 1250m, 1275m, 1290m, 1310m, 1360s, 1400s, 1450s, 1495s, 1640m, 2885m, 2985s, 3080m. ^{13}C NMR (CDCl_3/TMS): 78.6 (C-1); 24.3 (C-2); 68.8 (C-4); 27.3 (C-5); 48.3 (C-6); 117.6 (C-7); 134.0 (C-8); 147.0 (C-9); 124.9 (C-10, C-14); 127.9 (C-11, C-13); 126.4 (C-12). MS (DEI): m/z (%) $M + 1$, 231 (7.2); M^+ , 230 (1.4); $M - \text{allyl}$, 189 (27.7); $M + 1 - \text{BC}_3\text{H}_6\text{O}_2$, 145 (100.0).

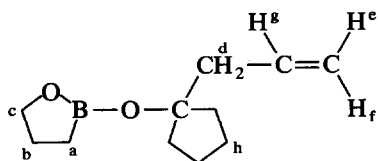


Anal. Found: B, 4.87; C, 72.60; H, 8.75. $\text{BC}_{14}\text{H}_{19}\text{O}_2$ calcd.: B, 4.70; C, 73.08; H, 8.32%.

3.1.5. 2-[1-(2-Propenyl)cyclopentyloxy]- (IIIe) and 2-[1-(2-propenyl)cyclohexyloxy]-1,2-oxaborolane (IIIg)

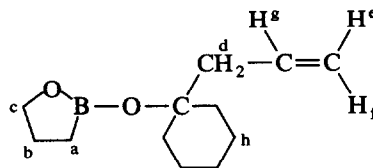
Starting from cyclopentanone or cyclohexanone and heating at 160°C for 3 h, IIIe or IIIg was obtained in an analogous way.

IIIe: Yield, 86.8%; b.p. $75\text{--}76^\circ\text{C}/4.6$ mbar, n_D^{20} 1.4681. ^1H NMR (CCl_4): δ 0.62 (t, 2H, $J = 7.2$, a); 1.51, 1.61 (m, 10H, h,b); 2.31 (d, 2H, $J = 6.3$, d); 3.77 (t, 2H, $J = 6.8$, c); 4.67, 4.86, 5.58 (m, 3H, e,f,g). IR (film): ν (cm^{-1}) 665m, 860w, 885w, 915s, 985s, 1030s, 1130m, 1190s, 1250s, 1305s, 1315s, 1360s, 1400s, 1450s, 1490s, 1640m, 2865s, 2945s, 3070m.



Anal. Found: B, 5.65; C, 67.39; H, 10.18. $\text{BC}_{11}\text{H}_{19}\text{O}_2$ calcd.: B, 5.57; C, 68.08; H, 9.87%.

IIIg: Yield, 65.4%; b.p. $87\text{--}88^\circ\text{C}/4$ mbar, n_D^{20} 1.4721. ^1H NMR (CCl_4): δ 0.72 (t, 2H, $J = 7.2$, a); 1.27 (m, 10H, h); 1.69 (q, 2H, $J = 7.6$, b); 2.27 (d, 2H, $J = 6.3$, d); 3.84 (t, 2H, $J = 6.8$, c); 4.74, 4.95, 5.63 (m, 3H, e,f,g). IR (film): ν (cm^{-1}) 670m, 680m, 730w, 780w, 825w, 865w, 880w, 915s, 970s, 995s, 1030m, 1145m, 1165m, 1195s, 1250s, 1300s, 1310m, 1365s, 1405s, 1460s, 1495s, 1645m, 2865s, 2930s, 3085m. MS (DEI): m/z (%) $M - \text{allyl}$, 167 (2.2); $M + 1 - \text{BC}_3\text{H}_6\text{O}_2$, 124 (66.3); 44 (100.0).



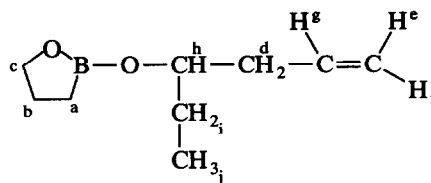
Anal. Found: B, 5.12; C, 68.85; H, 10.60. $\text{BC}_{12}\text{H}_{21}\text{O}_2$ calcd.: B, 5.19; C, 69.26; H, 10.17%.

3.2. General procedure for allylboration of aldehydes with II

Aldehyde (70 mmol) was added slowly to **II** (7.70 g, 70 mmol) over 30 min at 0°C . After addition, stirring was continued for another 2 h at room temperature. Then distillation under vacuum gave the product (**IIIg-i**). Redistillation provided the analytical sample for determination of physical, microanalytical and spectroscopic data.

3.2.1. 2-(5-Hexen-3-yloxy)-1,2-oxaborolane (IIIg)

Yield, 84.5%; b.p. $105\text{--}106^\circ\text{C}/76$ mbar, n_D^{20} 1.4473. ^1H NMR (CCl_4): δ 0.69, 0.72 (m, 5H, a,j); 1.33 (q, 2H, $J = 6.6$, i); 1.73 (q, 2H, $J = 7.8$, b); 2.06 (t, 2H, $J = 6.3$, d); 3.80 (t, 2H, $J = 6.8$, c); 3.93 (q, 1H, $J = 6.5$, h); 4.03, 4.94, 5.64 (m, 3H, e,f,g). IR (film): ν (cm^{-1}) 665m, 825w, 860w, 910s, 970s, 990s, 1015s, 1085m, 1110m, 1135m, 1165m, 1195s, 1250s, 1305s, 1345s, 1405–1420s, 1450–1460s, 1490s, 1645m, 2875s, 2930s, 2960s, 3080m. MS (DEI): m/z (%) $M + 1$, 169 (2.2); M^+ , 168 (0.8); $M - \text{allyl}$, 127 (24.3); $M + 1 - \text{BC}_3\text{H}_6\text{O}_2$, 84 (51.9); C_3H_7^+ , 43 (100.0).

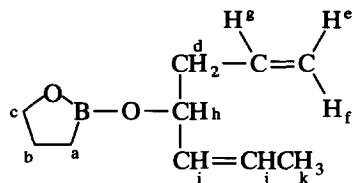


Anal. Found: B, 6.36; C, 63.68; H, 10.54. $\text{BC}_9\text{H}_{17}\text{O}_2$ calcd.: B, 6.43; C, 64.33; H, 10.20%.

3.2.2. 2-(1,6-Heptadien-4-yloxy)-1,2-oxaborolane (IIIh)

Yield, 86.5%; b.p. $86\text{--}87^\circ\text{C}/10$ mbar, n_D^{20} 1.4633. ^1H NMR (CCl_4): δ 0.65 (t, 2H, $J = 7.8$, a); 1.47 (d, 3H, $J = 6.4$, k); 1.66 (q, 2H, $J = 7.8$, b); 2.04 (t, 2H, $J = 7.8$, d); 3.74 (t, 2H, $J = 6.8$, c); 4.35 (m, 1H, h); 4.67, 4.88, 5.28, 5.33, 5.60 (m, 5H, e,f,i,j,g). IR (film): ν (cm^{-1}) 665m, 910s, 965s, 990s, 1020s, 1135m, 1200s, 1250s, 1310s, 1350s, 1415s, 1450s, 1490s, 1640m, 1675w, 2935s, 3075m. MS (DEI): m/z (%) $M + 1$, 181 (4.0);

M^+ , 180 (2.1); M - allyl, 139 (30.3); M - $BC_3H_6O_2$, 95 (100.0).

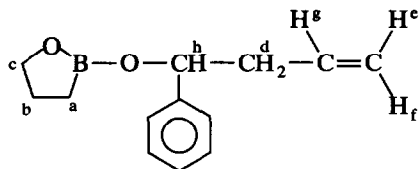


Anal. Found: B, 5.93; C, 66.44; H, 10.09. $BC_{10}H_{17}O_2$ calcd.: B, 6.00; C, 66.71; H, 9.52%.

3.2.3. 2-(1-Phenyl-3-buten-1-yloxy)-1,2-oxaborolane (IIIi)

Yield: 90.0%; b.p. 114–116°C/5 mbar, n_D^{20} 1.5167.

1H NMR (CCl_4): δ 0.76 (t, 2H, $J = 7.8$, a); 1.75 (q, 2H, $J = 7.4$, b); 2.37 (t, 2H, $J = 6.6$, d); 3.86 (t, 2H, $J = 6.8$, c); 4.77, 4.98, 5.05, 5.57 (m, 4H, e,h,f,g); 7.13 (m, 5H, Ph). IR (film): ν (cm^{-1}) 657w, 692s, 748m, 904m, 980s, 1012m, 1045m, 1125m, 1190s, 1242s, 1343s, 1400s, 1483s, 1595m, 1632m, 1700w, 1810w, 1862w, 1940w, 2845s, 2900s, 2985m, 3030m.



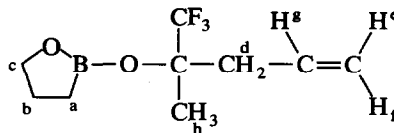
Anal. Found: B, 5.09. $BC_{13}H_{17}O_2$ calcd.: B, 5.00%.

3.3. 2-(1,1,1-Trifluoro-2-methyl-4-penten-2-yloxy)-1,2-oxaborolane (IIIj)

A 25 ml dry, nitrogen-filled round bottomed flask equipped with a side arm capped with a rubber septum, a magnetic stirring bar, a condenser with a dry-ice head and a connecting tube attached to a bubbler, was charged with 6.7 g (60 mmol) of **II**. The flask was immersed in a dry ice-alcohol cooling bath. Then 9.16 g (80 mmol) of trifluoroacetone was added dropwise slowly at low temperature with the aid of a cooling syringe. Stirring was continued for 2 h at room temperature after removing from the cooling bath. Then the mixture was heated at 100°C for a further hour. Distillation under vacuum gave 9.35 g (70.2%) of **IIIj**, b.p. 63–65°C/20 mbar, n_D^{20} 1.4030, d_4^{20} 1.1180.

1H NMR (CCl_4): δ 0.77 (t, 2H, $J = 8$, a); 1.33 (s, 3H, h); 1.73 (q, 2H, $J = 7.6$, b); 2.50 (d, 2H, $J = 6.4$, d); 3.93 (t, 2H, $J = 6.8$, c); 4.90, 5.06, 5.60 (m, 3H, e,f,g). ^{19}F NMR (CCl_4): δ 2.4 (upfield from TFA). IR (film): ν (cm^{-1}) 657w, 692s, 748m, 904m, 980s, 1012m, 1045m, 1125m, 1190s, 1242s, 1343s, 1400s, 1483s, 1595m, 1632m, 1700w, 1810w, 1862w, 1940w, 2845s, 2900s, 2985m, 3030m. MS (DEI): m/z (%) $M + 1$, 223 (4.2);

M^+ , 222 (1.7); M - allyl, 181 (0.8); M - $BC_3H_6O_2$, 137 (3.4), 43 (100.0).



Anal. Found: B, 4.65; C, 48.73; H, 6.40. $BC_9H_{14}O_2F_3$ calcd.: B, 4.87; C, 48.69; H, 6.36%.

3.4. Deboronation of III with diethanolamine. General procedure

Into a dry, nitrogen-filled micro-distillation flask equipped with a side arm capped with a rubber septum, a magnetic stirring bar, and a Claisen head attached to an oil-bubbler was placed 10 mmol of **III** and 0.64 g (about 6 mmol) of diethanolamine. The mixture was stirred and heated at 120–160°C for 1–2 h to give the product (**IVa** and **IVj**) or distillation under vacuum gave the product (**IVb–IVi**). Redistillation or column chromatography provided the analytical samples.

4. Conclusion

2-Allyl-1,2-oxaborolane (**II**) is a novel allylborane (allyl-BCO, a cyclic borinate ester) and can react with ketones or aldehydes to give the adducts **III** under mild conditions. 2-Alkenoxy-1,2-oxaborolanes (**III**) can be isolated in the pure state and then converted into the corresponding homoallylic alcohols **IV**.

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