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Lithium Bromide/HBpin: A Mild and Effective Catalytic System for the Selective Hydroboration of Aldehydes and Ketones

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The catalytic hydroboration of aldehydes and ketones with HBpin was examined using simple and commercially available metal salts (Li, Na, and K). Among the tested salts, LiBr (0.5–1.0 mol%) was found to be an efficient catalyst for the hydroboration of various aldehydes and ketones at room temperature. Further, the chemoselective hydroboration of aldehydes over ketones was also demonstrated.

Keywords: Catalyzed hydroboration, Lithium bromide, Chemoselective reduction, Pinacolborane (HBpin)

Introduction

Reduction reactions are fundamental and commonly used chemical transformations in organic chemistry. For example, the preparation of alcohols via the reduction of aldehydes and ketones is a well-known reaction. Among the various methods available for this transformation, hydride reduction reactions are often favored in terms of their yields and selectivities.[1] In addition, the hydroboration of carbonyls/unsaturated hydrocarbons using a less active reductant is an important tool for the preparation of alkoxy/boronate esters as building blocks for functionalized alcohols and borylation reactions, in addition to coupling agents in cross-coupling reactions.[2]

Recently, numerous catalytic systems have been reported for the hydroboration of carbonyl groups and unsaturated hydrocarbons, with examples including transition metals, main group metals, lanthanides, and nonmetal complexes.[3] However, in addition to being sensitive to the conditions employed, these complexes often involve the use of toxic reagents. Furthermore, they are expensive and require the prepreparation of ligands. To overcome these issues, research into alternative catalytic approaches is ongoing, whereby the shift from complexes to commercial bench-top reagents has received attention from an environmental and economic standpoint.

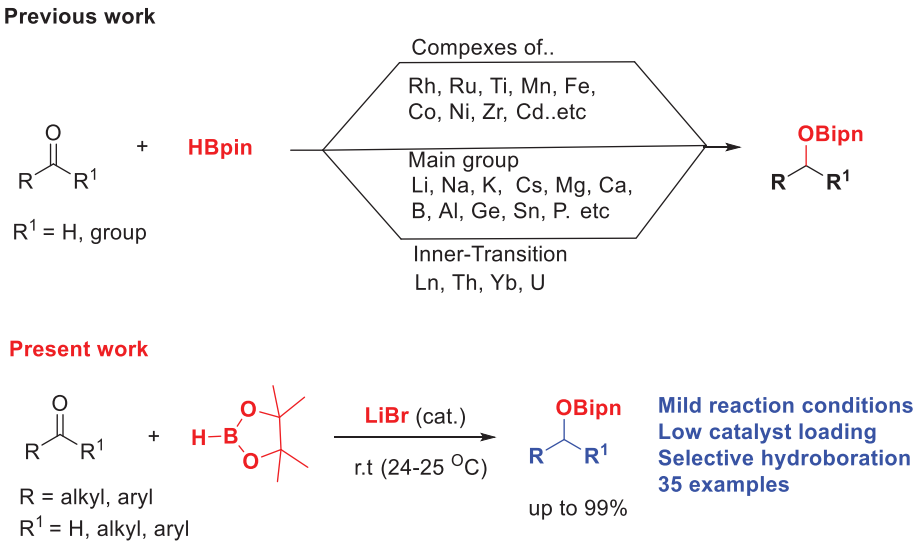
Based on the above points, Zhu's group recently reported protocols for the catalytic hydroboration of aldehydes, ketones, imines, and alkynes with n-BuLi.[4] In addition, Wu and co-workers demonstrated a general method for the catalytic hydroboration of aldehydes, ketones, alkynes, and alkenes with HBpin using powdered NaOH as a catalyst.[5] Recently, Hreczycho *et al.*, reported the potassium-fluoride mediated hydroboration of aldehydes and ketones.[6] More recently, our group demonstrated protocols for the catalytic hydroboration of carbonyls and unsaturated hydrocarbons using readily available reagents as catalysts.[7] Furthermore, the hydroboration of aldehydes and ketones has also been reported using a catalyst-free protocol under thermal conditions.[8,9] More specifically, Stachowiak *et al.*[8] described the hydroboration of aldehydes under catalyst-free conditions; however, their method was applicable only to aldehydes, and required high temperatures to achieve optimal conversions. Moreover, the Leung group reported the catalyst-free and solvent-free hydroboration of ketones at an elevated temperature.[9]

Thus, we herein present our study into the efficient and selective hydroboration of aldehydes and ketones with pinacolborane using LiBr as a catalyst at room temperature.

A literature study revealed the use of LiBr as a reagent and catalyst for the disproportionation and reduction of aldehydes, the formation of *N*-acetyl enamines and *N*-tosylimines, the selective cleavage of *N,N*-diprotected amines, and as an additive in dearomatizing cyclization reactions.[10] We therefore employ LiBr as a promoter for the hydroboration of aldehydes and ketones under mild reaction conditions (Scheme 1).

Results and Discussion

Initially, the influence of different alkali metal (Li, Na, K)-halides (F, Cl, Br, I) was examined to develop the catalytic system (Table 1). Thus, 1.0 mol% of the tested catalyst was treated with HBpin (2.0 eq), followed by reaction with 4-methoxybenzaldehyde. Among the catalysts examined, the hydroboration of 4-methoxybenzaldehyde using LiBr and LiI as catalysts afforded 99% conversion to the desired product after 1 h (entries 3–4, Table 1).



Scheme 1. Reported catalytic hydroboration reactions and the present LiBr-mediated hydroboration system.

Under the present protocol, LiBr was selected as the catalyst due to its lower cost compared to LiI. The optimized parameters developed using LiBr are presented in Table 2.

Subsequently, the catalyst loading, solvent, stoichiometry, and reaction time were examined. As shown in Table 2, the reaction of 4-methoxybenzaldehyde with 1.0 mol% LiBr and HBpin (2.0 eq) in THF (1 mL) over 1 h afforded the boronate ester in 99% yield (entry 1, Table 2). Interestingly, the reaction conversion was maintained upon decreasing the catalyst loading (1 to 0.5 mol%, entry 4, Table 2). However, under more dilute conditions (5 mL THF), 4% of unreacted aldehyde remained (entry 5, Table 2). In terms of the solvent system, neat conditions, toluene, diethyl ether, and methylene chloride gave moderate conversions along with 6–21% of the unreacted starting material (entry 3, entries 6–8, Table 2). In THF, 10% of the starting aldehyde remained in addition to the desired boronate when 1.5 eq HBpin and 0.5 mol% catalyst were employed over 6 h (entry 11, Table 2). Consequently, the optimal reaction parameters were as follows: LiBr (0.5 mol%), aldehyde (1.0 eq), and HBpin (2.0 eq), at room temperature for 1 h (entry 4, Table 2).

With the optimal conditions in hand, the scope of the catalytic hydroboration was explored using various aldehydes and ketones. Both aliphatic and aromatic/heteroaromatic aldehydes underwent hydroboration within an hour, giving excellent conversions to the desired boronates. This was followed by hydrolysis to the corresponding alcohols (Table 3). It was found that aldehydes bearing electron donating and electron withdrawing substituents were tolerated and afforded good conversions (Table 3, **2a–2i**). Importantly, alcohols **2f–2h** were obtained from their corresponding boronates in quantitative yields despite the effects of steric hindrance from the substituents (Table 3**, 2f–2h**). Moreover, hetero- and polyaromatic aldehydes also smoothly underwent the corresponding hydroboration reactions in quantitative yields (Table 3**, 2j** and **2k**). Furthermore, a conjugated aldehyde (*trans*-cinnamaldehyde) and ferrocene-carboxaldehyde afforded the corresponding products in excellent yields (Table 3**, 2l** and **2m**), and aliphatic aldehydes were also converted to the corresponding alcohols (Table 3**, 2n** and **2o**).

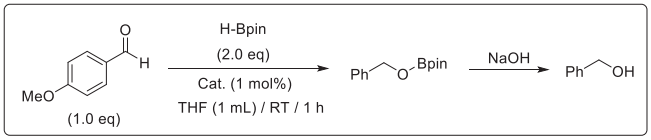
The catalytic hydroboration reaction was then carried out using ketone substrates with 1 mol% LiBr. As a result, the corresponding boronate esters and alcohols (following hydrolysis) were obtained in excellent yields (Table 4).

Examination of the ketone substrate scope showed that aromatic/hetero-aromatic, conjugated, aliphatic, and bulky ketones could be employed. Interestingly, irrespective of the electronic and steric factors of the substituents, all tested ketones underwent the hydroboration reaction with excellent conversions under ambient conditions. This is noteworthy when considering the harsh conditions employed by recently reported thermal systems.

Following the successful reductions of both aldehydes and ketones, the catalytic hydroboration reaction using LiBr was applied to other organic functional groups, including acyl chlorides, amides, nitriles, alkenes, alkynes, alkyl halides, and epoxy groups (5 mol% LiBr, 4 eq HBpin, THF, Table 5). It was found that only aliphatic and aromatic aldehydes and ketones underwent hydroboration, thereby indicating the chemoselective nature of the present system.

To determine the utility of the LiBr catalytic system for chemoselective reductions, we carried out the chemoselective hydroboration of a mixture of aldehydes and ketones using 0.5 mol% LiBr and 1.5 eq HBPin in THF over 1 h. Indeed, an excellent selectivity with 89% conversion was found for benzaldehyde in the presence of acetophenone (entry 1, Table 6). Similar results were obtained for electron-donating (methoxy) and electron-withdrawing (nitro) substituents (entries 2–3, Table 6). It

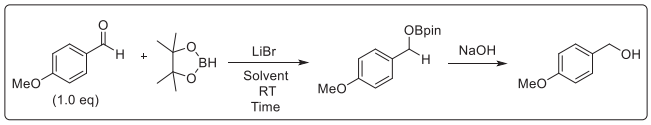
Table 1. Catalyst evaluation for the hydroboration of 4-methoxybenzaldehyde.



Entry	Cat.	Conv. (%)[a] (ROH)	Yield (%)[b] (S.M / ROH)
1	LiF	15	85 / 15
2	LiCl	90	10 / 90
3	LiBr	99	0 / 99
4	LiI	99	0 / 99
5	NaF	12	88 / 12
6	NaCl	12	88 / 12
7	NaBr	6	94 / 6
8	NaI	45	55 / 45
9	KF	9	91 / 9
10	KCl	12	88 / 12
11	KBr	15	85 / 15
12	KI	13	87 / 13

[a] Conversions were determined by the area ratio of GC.
[b] Yields were determined by GC using naphthalene as an internal standard.

Table 2. Optimization of the reaction conditions.



Entry	HBpin	LiBr	Solvent	Time	Conv. (%)[a] (ROH)	Yield (%)[b] (S.M / ROH)
1	2.0 eq	1.0 mol%	THF (1 mL)	1 h	99	0 / 99
2			THF (1 mL)	3 h	99	0 / 99
3	2.0 eq	0.5 mol%	neat	1 h	94	6 / 94
4			THF (1 mL)	1 h	99	0 / 99
5			THF (5 mL)	1 h	96	4 / 96
6			Toluene (1 mL)	1 h	99	1 / 80
7			Ether (1 mL)	1 h	88	12 / 88
8			CH_2Cl_2 (1 mL)	1 h	85	15 / 85
9	1.5 eq	0.5 mol%	THF (1 mL)	1 h	78	21 / 79
10			THF (1 mL)	3 h	90	10 / 90
11			THF (1 mL)	6 h	90	10 / 90

[a] Conversions were determined by the area ratio of GC.
[b] Yields were determined by GC using naphthalene as an internal standard.

was also found that aliphatic aldehydes selectively underwent the hydroboration reaction in the presence of aromatic/aliphatic ketones, with a good chemoselectivity and yield being obtained in both cases (entries 5–6, Table 6). A high chemoselectivity was also obtained for molecules containing more than one reducible functional group (Table 6 and Scheme 2).

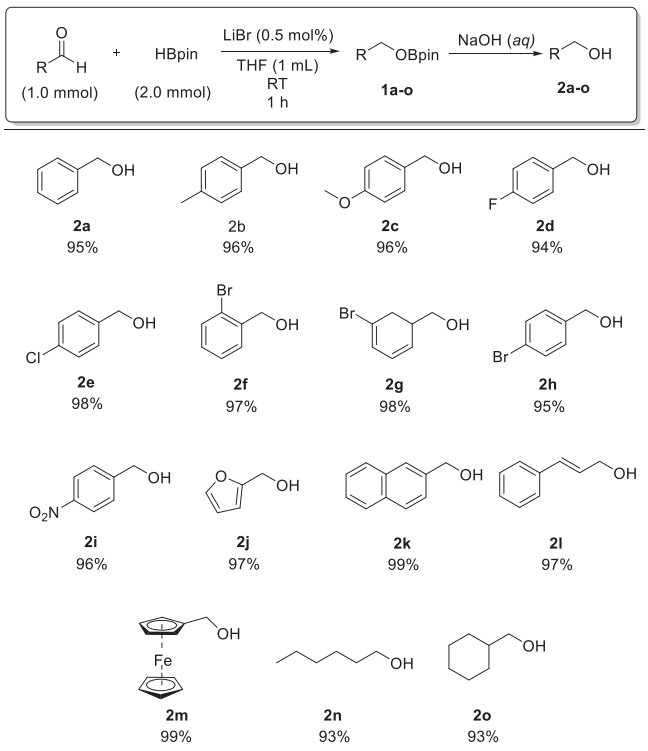
Subsequently, density functional theory (DFT) calculations were performed to determine the possible reaction pathway for the LiBr-catalyzed hydroboration of aldehydes. The energy profile for the reaction was presented in Scheme 3. Based on this computed reaction energy profile, the reaction pathway for the LiBr-catalyzed hydroboration of acetaldehyde was investigated using DFT calculations at the M06-2X/6–31 + G(d,p) level of theory. In this mechanism, HBpin initially reacts with the LiBr catalyst to form the intermediate complex **INT1**. The binding of aldehyde to **INT1** results in the formation of **INT2** which in turn undergoes a unimolecular transformation into **INT3** through a hexagonal ring transition state **TS1**. Then **INT3** goes through another unimolecular transformation to give **INT4** via a four-membered ring transition state **TS2**. Finally, the catalytic cycle is fulfilled when **INT4** reacts with a second molecule of HBpin to regenerate **INT1** and produce the boronate ester through a ligand exchange reaction. Based on the energy profile presented in Scheme 3, a plausible mechanism is summarized in Scheme 4.

In summary, we successfully identified an economic and convenient method for the selective hydroboration of aldehydes and ketones at room temperature. Among the tested metal halides, LiBr was found to be superior in terms of the conversions and yields. In addition, a variety of aldehydes and ketones containing a wide range of substituents were tolerated, and the hydroboration proceeded efficiently in the presence of LiBr. Furthermore, we demonstrated that the present system enables the chemoselective catalytic hydroboration of aldehydes over ketones. Overall, the mildness of the catalyst and the greater selectivity with almost quantitative conversions rendered this protocol useful in the synthesis of alcohols via catalytic hydroboration.

Experimental

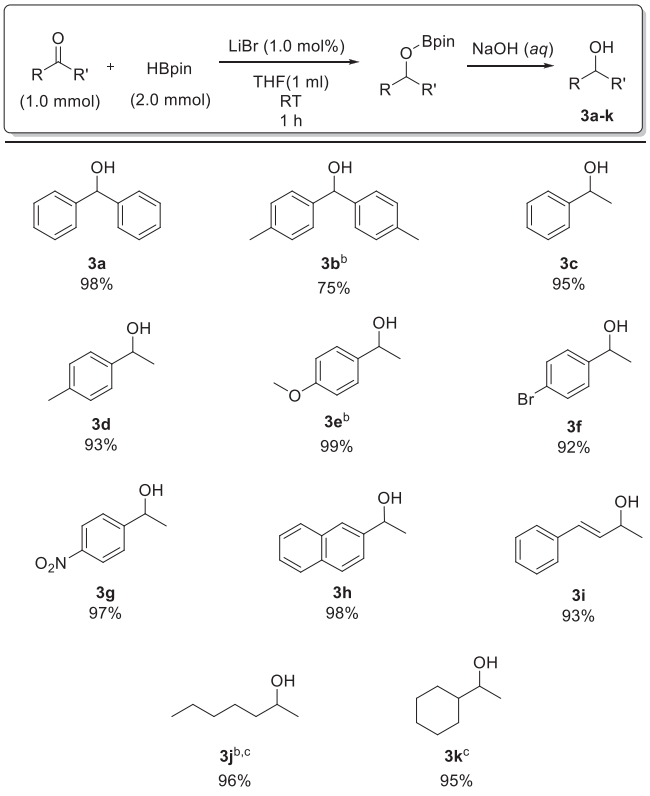
General. Prior to use, all glassware was dried thoroughly in an oven, assembled hot, and cooled under a stream of dry nitrogen. All reactions and manipulations of air- and moisture-sensitive materials were carried out using standard techniques for the handling of such materials. All chemicals were commercial products of the highest purity which were further purified before use using standard methods. HBpin, aldehydes, ketones, alkenes were purchased from Aldrich

Table 3. Catalytic hydroboration reactions of aldehydes using LiBr.[a]



[a] Isolated yields after silica column chromatography.

Table 4. Catalytic hydroboration reactions of ketones with LiBr[a].



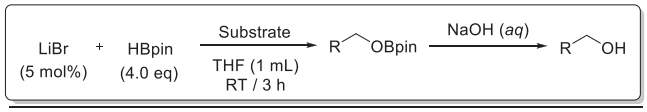
[a] Isolated yields after silica column chromatography.
[b] LiBr used 5.0 mol% without solvent.
[c] Yields were determined by NMR.

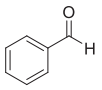
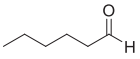
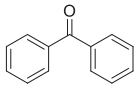
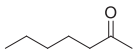
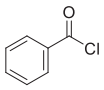
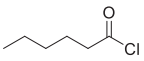
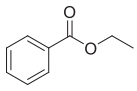
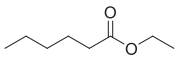
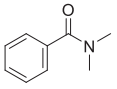
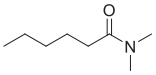
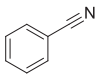
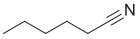
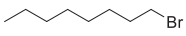
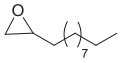
Chemical Company, Alfa Aesar, and Tokyo Chemical Industry Company (TCI, Tokyo, Japan). ^{1}H NMR spectra were measured at 400 MHz with $CDCl_3$ as a solvent at ambient temperature unless otherwise indicated, and the chemical shifts were recorded in parts per million downfield from tetramethylsilane (δ = 0 ppm) or based on residual $CDCl_3$ (δ = 7.26 ppm) as the internal standard. ^{13}C NMR spectra were recorded at 100 MHz with $CDCl_3$ as a solvent and referenced to the central line of the solvent (δ = 77.0 ppm). The coupling constants (J) are reported in hertz. Analytical thin-layer chromatography (TLC) was performed on glass precoated with silica gel (Merck, silica gel 60 F254). Column chromatography was carried out using 70–230 mesh silica gel (Merck) at normal pressure. GC analyses were performed on a Younglin Acme 6100M and 6500 GC FID chromatography system, using an HP-5 capillary column (30 m). All GC yields were determined with the use of naphthalene as the internal standard and the authentic sample.

General Procedure for the Hydroboration of Aldehydes (2a–o). A dry and argon-flushed flask, equipped with a magnetic stirring bar was charged with lithium bromide (0.0004 g, 0.5 mol%), benzaldehyde (0.1 mL, 1.0 mmol), and THF (1 mL) at room temperature. To this, pinacolborane (0.29 mL, 2.0 mmol) was added dropwise at room temperature and stirred for 1 h. The reaction was terminated by the addition of water (0.1 mL). The conversion of the boronate ester was confirmed by GC and the crude mixture was hydrolyzed to the alcohol by the addition of a 1 *N* aqueous NaOH solution (3 mL). After stirring for 30 min, diethyl ether (5 mL) and NaCl were added until the solution became supersaturated. The crude mixture was extracted with ethyl acetate and the combined organic layers were dried over anhydrous $MgSO_4$. After filtration, the solvents were evaporated under reduced pressure and the mixed residue was purified by silica gel column chromatography.

General Procedure for the Hydroboration of Ketones (3a–k). A dry and argon-flushed flask, equipped with a magnetic stirring bar was charged with lithium bromide (0.0009 g, 1.0 mol%), acetophenone (0.12 mL, 1.0 mmol), and THF (1 mL) at room temperature. To this, pinacolborane (0.29 mL, 2.0 mmol) was added dropwise at room temperature and stirred for 1 h. The reaction was terminated by the addition of water (0.1 mL). The conversion of the boronate ester was confirmed by ^{1}H NMR and the crude mixture was hydrolyzed to the alcohol by the

Table 5. Reactivity screening of representative organic functional groups.[a]



Entry	Substrate	Yield (%)[a]
1		98
2		96
3[b]		99
4		99
5		99
6		No reaction
7		No reaction
8		No reaction
9		No reaction
10		No reaction
11		No reaction
12		No reaction
13		No reaction
14		No reaction
15		No reaction
16		No reaction
17		No reaction

[a] Yields were determined by GC.

[b] Yields were determined by NMR.

addition of a 1 *N* aqueous NaOH solution (3 mL). After stirring for 30 min, NaCl was added until the solution became supersaturated. The crude mixture was extracted with ethyl acetate and the combined organic layers were dried over anhydrous $MgSO_4$. After filtration, the solvents were evaporated under reduced pressure and mixed residue was purified by silica gel column chromatography.

General Procedure for the Chemoselective Hydroboration of Aldehydes in the Presence of Ketones. A dry and argon-flushed flask, equipped with a magnetic stirring bar was charged with lithium bromide (0.0004 g, 0.5 mol%), benzaldehyde (0.10 mL, 1.0 mmol), acetophenone (0.12 mL, 1.0 mmol), and THF (1 mL) at room temperature (Table 6). To this, pinacolborane (0.22 mL, 1.5 mmol) was added at room temperature and stirred for 1 h. The reaction was terminated by the addition of water (0.1 mL). The crude mixture was hydrolyzed to the alcohol by the addition of a 1 *N* aqueous NaOH solution (3 mL). After stirring for 30 min, diethyl ether (5 mL) and NaCl were added until solution became supersaturated. The organic layer was extracted with ethyl acetate, the combined organic layers were dried over anhydrous $MgSO_4$, and the yields were calculated using GC.

4,4,5,5-Tetramethyl-2-(benzyloxy)-1,3,2-dioxaborolane (1a). Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37–7.30 (m, 4H), 7.27–7.24 (m, 1H), 4.92 (s, 2H), 1.25 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.30, 128.39, 127.46, 126.81, 83.08, 66.75, 24.71. NMR data were in accordance with reported literature.[11]

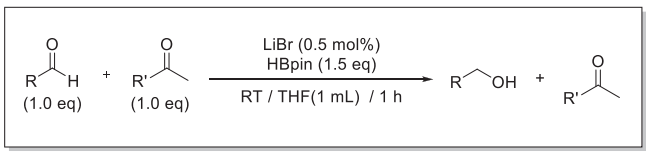
4,4,5,5-Tetramethyl-2-((4-methylbenzyl)oxy)-1,3,2-dioxaborolane (1b). Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.23 (d, J = 8.0 Hz, 2H), 7.13 (d, J = 8.0 Hz, 2H), 4.87 (s, 2H), 2.32 (s, 3H), 1.25 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.12, 136.32, 129.06, 126.94, 83.01, 66.67, 24.71, 21.25. NMR data were in accordance with reported literature.[11]

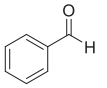
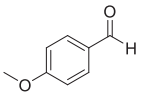
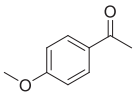
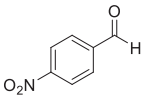
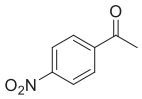
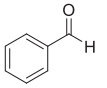
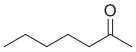
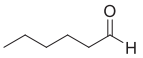
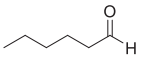
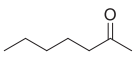
2-((4-Methoxybenzyl)oxy)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1c). Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (d, J = 8.4 Hz, 2H), 6.85 (d, J = 8.6 Hz, 2H), 4.84 (s, 2H), 3.79 (s, 3H), 1.25 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.10, 131.55, 128.62, 113.75, 83.00, 66.52, 55.37, 24.63. NMR data were in accordance with reported literature.[11]

2-((4-Fluorobenzyl)oxy)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1d). Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (dd, J = 8.3, 5.5 Hz, 2H), 7.00 (t, J = 8.7 Hz, 2H), 4.86 (s, 2H), 1.25 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.29 (d, J = 246.4 Hz), 135.05 (d, J = 3.0 Hz), 128.72 (d, J = 8.1 Hz), 115.21(d, J = 22.2 Hz), 83.15, 66.15, 24.70. NMR data were in accordance with reported literature.[11]

2-((4-Chlorobenzyl)oxy)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1e). Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32–7.25 (m, 4H), 4.87 (s, 2H), 1.25 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.69, 133.10, 128.44,

Table 6. Chemoselective catalytic hydroboration of aldehydes over ketones.



Entry	Aldehyde	Ketone	Yield (%)[a] Aldehyde SM / ROH	Yield (%)[a] Ketone SM / ROH
1			9 / 89	98 / 0
2[b,c]			10 / 90	99 / 0
3[b,d]			2 / 98	97 / 0
4			8 / 90	97 / 3
5			4 / 96	96 / 0
6			5 / 95	96 / 4

[a] Yields were determined by GC using naphthalene as an internal standard.
[b] Yields were determined by NMR using 1,3,5-trimethoxybenzena as an internal standard.
[c] Using HBpin (2.0 eq).
[d] Using HBpin (1.1 eq).

128.10, 83.11, 65.95, 24.61. NMR data were in accordance with reported literature.[12]

2-((2-Bromobenzyl)oxy)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1f). Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 (dd, J = 7.9, 1.1 Hz, 2H), 7.30 (td, J = 7.6, 1.2 Hz, 1H), 7.12 (td, J = 7.7, 1.7 Hz, 1H), 4.97 (s, 2H), 1.26 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.43, 132.35, 128.70, 127.88, 127.45, 121.61, 83.26, 77.43, 24.71. NMR data were in accordance with reported literature.[8]

2-((3-Bromobenzyl)oxy)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1g). Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (s, 1H), 7.38 (d, J = 7.8 Hz, 1H), 7.25–7.16 (m, 2H), 4.88 (s, 2H), 1.26 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.57, 130.52, 129.97, 129.80, 125.23, 122.54, 83.27, 65.92, 24.70. NMR data were in accordance with reported literature.[13]

2-((4-Bromobenzyl)oxy)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1h). Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.44 (dd, J = 8.4, 1.8 Hz, 2H), 7.23–7.18 (m, 2H), 4.86 (s, 2H), 1.25 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.30, 131.48, 128.50, 121.30, 83.22, 66.06, 24.70. NMR data were in accordance with reported literature.[12]

4,4,5,5-Tetramethyl-2-((4-nitrobenzyl)oxy)-1,3,2-dioxaborolane (1i). Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, J = 8.7 Hz, 2H), 7.48 (d, J = 8.3 Hz, 2H), 5.00 (s, 2H), 1.24 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.30, 146.70, 126.93, 123.68, 83.48, 65.62, 24.68. NMR data were in accordance with reported literature.[12]

2-(Furan-2-ylmethoxy)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1j). Yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (dd, J = 1.7, 0.9 Hz, 1H), 6.31–6.27 (m, 2H), 4.81 (s, 2H), 1.24 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 152.53, 142.56, 110.34, 108.41, 83.17, 59.28, 24.68. NMR data were in accordance with reported literature.[8]

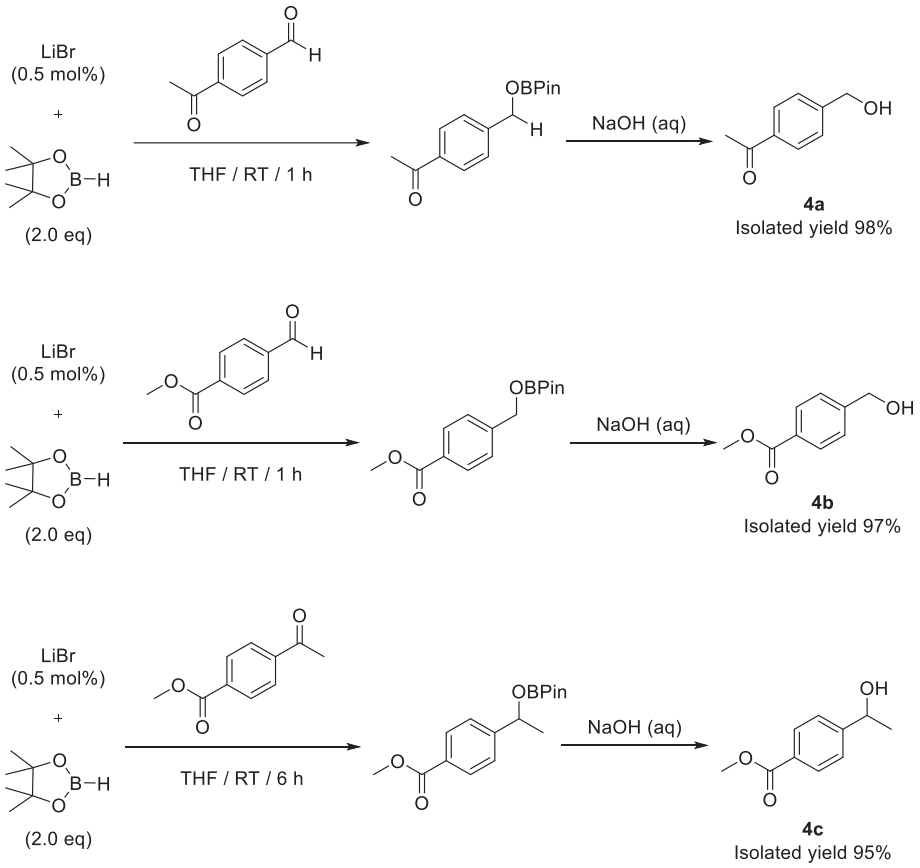
4,4,5,5-Tetramethyl-2-(naphthalen-2-ylmethoxy)-1,3,2-dioxaborolane (1k). Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.87–7.76 (m, 4H), 7.50–7.40 (m, 3H), 5.10 (s, 2H), 1.26 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.79, 133.42, 132.92, 128.09, 128.02, 127.77, 126.13, 125.83, 125.24, 124.96, 83.17, 66.84, 24.74. NMR data were in accordance with reported literature.[11]

2-(Cinnamyloxy)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1l). Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, J = 7.1 Hz, 2H), 7.30 (t, J = 7.5 Hz, 2H), 7.27–7.16 (m, 2H), 6.61 (d, J = 15.9 Hz, 1H), 6.27 (dt, J = 15.9, 5.3 Hz, 1H), 4.53 (dd, J = 5.3, 1.6 Hz, 2H), 1.26 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.96, 130.68, 128.62, 127.60, 126.88, 126.54, 83.04, 65.36, 24.71. NMR data were in accordance with reported literature.[11]

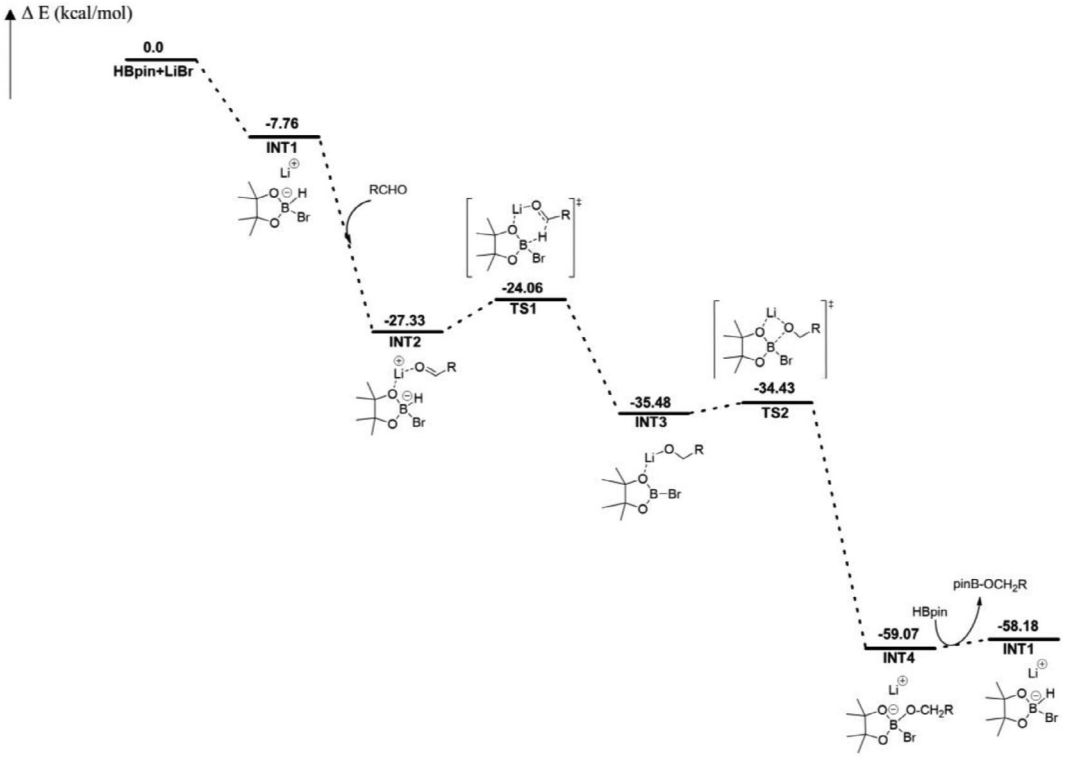
2-(Ferrocenylmethoxy)pinacolborane (1m). Yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.63 (s, 2H), 4.23 (t, J = 1.8 Hz, 2H), 4.20–4.07 (m, 7H), 1.25 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 85.46, 82.90, 68.52, 68.24, 63.26, 24.75. NMR data were in accordance with reported literature.[11]

2-(Hexyloxy)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1n). Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.82 (t, J = 6.6 Hz, 2H), 1.58–1.50 (m, 2H), 1.43–1.26 (m, 6H), 1.24 (s, 12H), 0.86 (t, J = 6.7 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 82.68, 65.10, 31.62, 31.51, 25.37, 24.64, 22.72, 14.14. NMR data were in accordance with reported literature.[14]

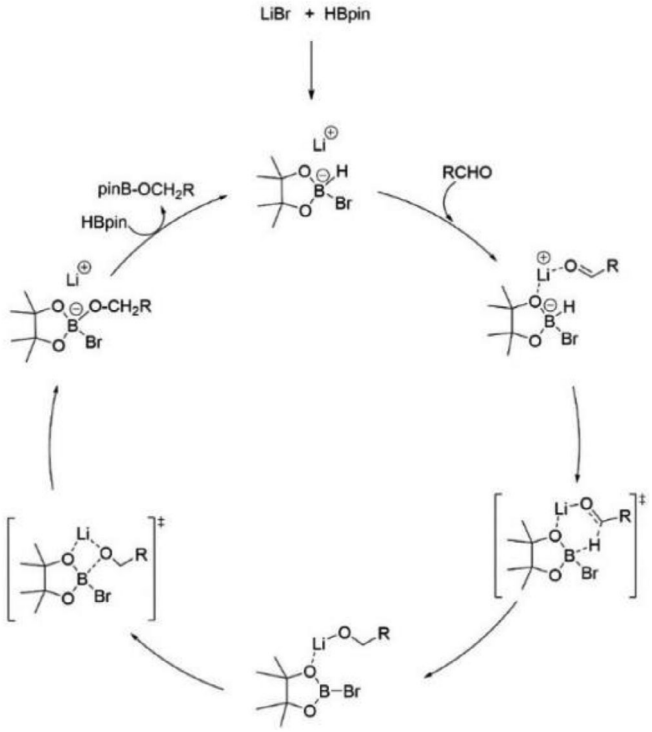
2-(Cyclohexylmethoxy)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1o). Colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.62 (d, J = 6.4 Hz, 2H), 1.72–1.60 (m, 5H), 1.54–1.44 (m, 1H), 1.22 (s, 12H), 1.19–1.03 (m, 3H), 0.92 (td, J = 11.8, 3.1 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ



Scheme 2. Chemoselective catalytic hydroboration of compounds containing more than one reducible group.



Scheme 3. Free energy profile (in kcal/mol) for the LiBr catalyzed hydroboration of aldehydes (R = CH_3).



Scheme 4. Plausible reaction mechanism for the lithium bromide-catalyzed hydroboration of aldehydes.

82.67, 70.46, 39.38, 29.40, 26.62, 25.86, 24.65. NMR data were in accordance with reported literature.[11]

Phenylmethanol (2a). Colorless oil (103 mg, 95% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, J = 4.4 Hz, 4H), 7.33–7.26 (m, 1H), 4.69 (s, 2H), 1.98 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ140.94, 128.69, 127.79, 127.11, 65.50. NMR data were in accordance with reported literature.[15]

***p*-Tolylmethanol (2b).** White solid (117 mg, 96% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 (d, J = 8.0 Hz, 2H), 7.17 (d, J = 7.8 Hz, 2H), 4.64 (s, 2H), 2.34 (s, 3H), 1.57 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.99, 137.55, 129.37, 127.24, 65.41, 21.26. NMR data were in accordance with reported literature.[16]

(4-Methoxyphenyl)methanol (2c). White solid (133 mg, 96% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (d, J = 8.6 Hz, 2H), 6.89 (d, J = 8.6 Hz, 2H), 4.61 (s, 2H), 3.80 (s, 3H), 1.59 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.32, 133.19, 128.78, 114.05, 65.18, 55.40. NMR data were in accordance with reported literature.[15]

(4-Fluorophenyl)methanol (2d). White solid (119 mg, 94% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38–7.28 (m, 1H), 7.08–6.99 (m, 1H), 4.66 (d, J = 4.5 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.64, 161.20, 136.67, 136.64, 128.91, 128.83, 115.61, 115.40, 64.79. NMR data were in accordance with reported literature.[15]

(4-Chlorophenyl)methanol (2e). White solid (140 mg, 98% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38–7.25 (m, 4H), 4.66 (s, 2H), 1.71 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.33, 133.46, 128.79, 128.38, 64.68. NMR data were in accordance with reported literature.[15]

(2-Bromophenyl)methanol (2f). White solid (181 mg, 97% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (dd, J = 7.9, 1.1 Hz, 1H), 7.47 (dd, J = 7.6, 1.6 Hz, 1H), 7.33 (td, J = 7.5, 1.1 Hz, 1H), 7.16 (td, J = 7.7, 1.7 Hz, 1H), 4.74 (s, 2H), 1.98 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.80, 132.71, 129.25, 129.03, 127.77, 122.70, 65.23. NMR data were in accordance with reported literature.[15]

(3-Bromophenyl)methanol (2g). Colorless oil (183 mg, 98% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (s, 1H), 7.38 (d, J = 7.3 Hz, 1H), 7.25–7.16 (m, 2H), 4.88 (s, 2H), 1.26 (s, 12H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.18, 130.76, 130.23, 129.99, 125.43, 122.75, 64.60. NMR data were in accordance with reported literature.[15]

(4-Bromophenyl)methanol (2h). White solid (178 mg, 95% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52–7.43 (m, 2H), 7.23 (d, J = 8.5 Hz, 2H), 4.66 (s, 2H), 1.67 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 139.84, 131.74, 128.70, 121.56, 64.71. NMR data were in accordance with reported literature.[15]

(4-Nitrophenyl)methanol (2i). Pale yellow solid (147 mg, 96% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21 (d, J = 8.7 Hz, 2H), 7.53 (d, J = 8.4 Hz, 2H), 4.83 (s, 2H), 1.90 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 148.19, 147.41, 127.10, 123.86, 64.14. NMR data were in accordance with reported literature.[16]

Furan-2-ylmethanol (2j). Pale yellow oil (95 mg, 97% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41–7.38 (m, 1H), 6.36–6.27 (m, 2H), 4.61 (s, 2H), 1.42 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 154.04, 142.73, 110.47, 107.91, 77.43, 77.12, 76.80, 57.62. NMR data were in accordance with reported literature.[15]

Naphthalen-2-ylmethanol (2k). White solid (157 mg, 99% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.88–7.78 (m, 4H), 7.53–7.44 (m, 3H), 4.86 (s, 2H), 1.73 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.38, 133.46, 133.03, 128.47, 127.99, 127.82, 126.31, 126.03, 125.55, 125.27, 65.63. NMR data were in accordance with reported literature.[15]

(*E*)-3-Phenylprop-2-en-1-ol (2l). White solid (130 mg, 97% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (dt, J = 8.0, 1.8 Hz, 2H), 7.31 (td, J = 6.7, 6.1, 1.7 Hz, 2H), 7.26–7.21 (m, 1H), 6.61 (d, J = 15.9 Hz, 1H), 6.36 (dt, J = 15.9, 5.7 Hz, 1H), 4.32 (d, J = 7.1 Hz, 2H), 1.48 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.75, 131.27, 128.70, 128.57, 127.82, 126.57, 63.87. NMR data were in accordance with reported literature.[17]

Ferrocenemethanol (2m). Yellow solid (214 mg, 99% yield);

^{1}H NMR (400 MHz, $CDCl_3$) δ 4.31 (d, J = 4.4 Hz, 2H), 4.25 (s, 2H), 4.22–4.11 (m, 7H), 1.49 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 88.71, 68.48, 68.04, 60.85. NMR data were in accordance with reported literature.[17]

Hexan-1-ol (2n). Colorless oil (95 mg, 93% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.63 (t, J = 6.6 Hz, 2H),

1.60–1.51 (m, 2H), 1.36–1.24 (m, 6H), 0.93–0.80 (m, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 63.21, 32.87, 31.72, 25.51, 22.73, 14.13. NMR data were in accordance with reported literature.[18]

Cyclohexylmethanol (2o). Colorless oil (106 mg, 93% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.43 (d, J = 6.4 Hz, 2H), 1.77–1.71 (m, 3H), 1.69–1.62 (m, 1H), 1.53–1.42 (m, 1H), 1.30–1.08 (m, 4H), 0.92 (qd, J = 13.6, 12.8, 3.5 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 68.90, 40.58, 29.64, 26.67, 25.92. NMR data were in accordance with reported literature.[15]

Diphenylmethanol (3a). White solid (180 mg, 97% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41–7.36 (m, 4H), 7.35–7.30 (m, 4H), 7.28–7.25 (m, 2H), 5.85 (s, 1H), 2.19 (s, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.89, 128.62, 127.70, 126.64, 76.37. NMR data were in accordance with reported literature.[11]

di-*p*-Tolylmethanol (3b). White solid (159 mg, 75% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27–7.25 (m, 2H), 7.24–7.23 (m, 2H), 7.13 (d, J = 7.9 Hz, 4H), 5.79 (s, 1H), 2.31 (s, 6H), 2.09 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 141.19, 137.27, 129.25, 126.52, 76.04, 21.20. NMR data were in accordance with reported literature.[19]

1-Phenylethan-1-ol (3c). Colorless oil (116 mg, 95% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42–7.31 (m, 4H), 7.30–7.24 (m, 1H), 4.88 (q, J = 4.3 Hz, 1H), 1.95 (bs, 1H), 1.49 (d, J = 6.5 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.92, 128.61, 127.59, 125.50, 70.52, 25.26. NMR data were in accordance with reported literature.[11]

1-(*p*-Tolyl)ethan-1-ol (3d). Colorless oil (127 mg, 93% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (d, J = 8.2 Hz, 2H), 7.16 (d, J = 7.9 Hz, 2H), 4.86 (q, J = 6.4 Hz, 1H), 2.34 (s, 3H), 1.82 (bs, 1H), 1.48 (d, J = 6.5 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 142.97, 137.27, 129.27, 125.46, 70.36, 25.19, 21.20. NMR data were in accordance with reported literature.[11]

1-(4-Methoxyphenyl)ethan-1-ol (3e). Colorless oil (150 mg, 99% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, J = 8.3 Hz, 2H), 6.87 (d, J = 8.7 Hz, 2H), 4.83 (q, J = 6.4 Hz, 1H), 3.79 (s, 3H), 2.02 (bs, 1H), 1.46 (d, J = 6.5 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.04, 138.12, 126.78, 113.92, 70.04, 55.38, 25.12. NMR data were in accordance with reported literature.[11]

1-(4-Bromophenyl)ethan-1-ol (3f). White solid (185 mg, 92% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49–7.42 (m, 2H), 7.25–7.18 (m, 2H), 4.84 (q, J = 6.5 Hz, 1H), 1.98 (bs, 1H), 1.45 (d, J = 6.5 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.86, 131.65, 127.26, 121.26, 69.88, 25.35. NMR data were in accordance with reported literature.[11]

1-(4-Nitrophenyl)ethan-1-ol (3g). Colorless oil (162 mg, 97% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, J = 8.8 Hz, 2H), 7.52 (d, J = 8.7 Hz, 2H), 5.00 (q, J = 6.5 Hz, 1H), 2.20 (bs, 1H), 1.49 (d, J = 6.5 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 153.22, 147.23, 126.23, 123.86, 69.59, 25.60. NMR data were in accordance with reported literature.[11]

1-(Naphthalen-2-yl)ethan-1-ol (3h). White solid (169 mg, 98% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85–7.80 (m, 4H), 7.52–7.43 (m, 3H), 5.07 (q, J = 6.5 Hz, 1H), 1.86 (bs, 1H), 1.58 (d, J = 6.5 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.27, 133.42, 133.02, 128.43, 128.04, 127.78, 126.26, 125.91, 123.91, 70.66, 25.25. NMR data were in accordance with reported literature.[20]

(*E*)-4-Phenylbut-3-en-2-ol (3i). White solid (138 mg, 93% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, J = 8.1 Hz, 2H), 7.31 (t, J = 7.5 Hz, 2H), 7.27–7.21 (m, 1H), 6.56 (d, J = 15.9 Hz, 1H), 6.26 (dd, J = 15.9, 6.4 Hz, 1H), 4.48 (q, J = 6.4 Hz, 1H), 1.95 (bs, 1H), 1.37 (d, J = 6.4 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.80, 133.69, 129.47, 128.71, 127.75, 126.58, 69.02, 23.52. NMR data were in accordance with reported literature.[21]

Heptan-2-ol (3j). Colorless oil (96% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.84–3.72 (m, 1H), 1.91 (bs, 1H), 1.44–1.37 (m, 2H), 1.32–1.25 (m, 6H), 1.19–1.15 (m, 3H), 0.88 (t, J = 6.4 Hz, 3H); NMR data were in accordance with reported literature.[22]

1-Cyclohexylethan-1-ol (3k). Colorless oil (95% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.58–3.49 (m, 1H), 1.93 (bs, 1H), 1.86–1.59 (m, 6H), 1.18–1.11 (m, 5H), 1.02–0.87 (m, 3H); NMR data were in accordance with reported literature.[23]

1-(4-(Hydroxymethyl)phenyl)ethan-1-one (4a). White solid (147 mg, 98% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99–7.92 (m, 2H), 7.49–7.42 (m, 2H), 4.77 (s, 2H), 2.60 (s, 3H), 1.23 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 197.95, 146.22, 136.46, 128.73, 126.72, 64.75, 26.78. NMR data were in accordance with reported literature.[24]

Methyl 4-(hydroxymethyl)benzoate (4b). White solid (161 mg, 97% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, J = 8.3 Hz, 2H), 7.45 (d, J = 8.1 Hz, 2H), 4.79 (s, 2H), 3.92 (s, 3H), 1.75 (bs, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ (ppm) 167.04, 146.01, 129.95, 129.42, 126.55, 64.81, 52.23. NMR data were in accordance with reported literature.[25]

Methyl 4-(1-hydroxyethyl)benzoate (4c). White solid (171 mg, 95% yield); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04–7.98 (m, 2H), 7.46–7.40 (m, 2H), 4.96 (q, J = 6.5 Hz, 1H), 3.90 (s, 3H), 1.49 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.03, 150.95, 129.95, 129.32, 125.37, 70.12, 52.20, 25.43. NMR data were in accordance with reported literature.[26]

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Supporting Information. Additional supporting information may be found online in the Supporting Information section at the end of the article.

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