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Short Communication

Rapid and green synthesis of phenols catalyzed by a deep eutectic mixture based on fluorinated alcohol in water

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ABSTRACT

A new deep eutectic mixture based on choline chloride and 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP) was prepared and was found to be effective for the rapid transformation of aryl/heteroaryl boronic acids to the corresponding phenols in water using hydrogen peroxide as the oxidant. Broad substrate compatibility, metal- and additive-free conditions as well as reusability of the catalyst made this procedure more environmentally benign.

1. Introduction

Phenol compounds and derivatives have been found in numerous natural products and they frequently serve as key synthetic intermediates for construction of more complex structures [1]. A routine method for preparation of phenols compounds involves either the nucleophilic substitution of activated aryl halides or metal-catalyzed transformations of diazoarenes [2,3]. However, these methods suffer from drawbacks such as harsh conditions or the incompatibilities of the substrates. In recent years, metal-catalyzed hydroxylation of aryl halides has emerged as an attractive alternative for the preparation of phenols and impressive progress has been achieved by several groups [4–10].

On the other hand, aromatic boronic acid derivatives are powerful synthons in organic chemistry due to the advantages such as non-toxic, stable to heat, air and moisture, and easy availability. Recent investigations also clearly showed that aromatic boronic acids could be transformed to phenols *via* copper-catalyzed hydroxylation or oxidative hydroxylation in the presence of a wide variety of catalysts or reagents. For example, Wang et al. has reported $CuSO_4$-catalyzed hydroxylation of aromatic boronic acids at room temperature, albeit using a stoichiometric strong base (KOH) and a nitrogen ligand [11]. The metal-free oxidative hydroxylation of aromatic boronic acids have also been achieved in various reaction system including aqueous H_2O_2 [12], H_2O_2-I_2 [13], H_2O_2-poly(N-vinylpyrrolidone) [14], H_2O_2-Amberlite IR-120 resin [15], *N*-oxides [16] and KOH/TBHP [17]. However, these methods have some disadvantages such as long reaction times, a large amount of oxidants or reagents, or the use of toxic chlorinated organic solvent. Thus, a rapid, metal- and additive-free, and eco-friendly protocol is still in demand.

More recently, deep eutectic solvents (DESs) have invoked enormous interest as a green and potential solvent or catalyst in organic reactions [18]. DESs are mainly prepared by combining a cationic salt with a hydrogen-bond donor. One of the most explicit examples is the mixing one mole of choline chloride (ChCl) with two moles of urea (with melting points of 247 °C and 133 °C, respectively), which results in a deep eutectic solvent with a room temperature melting point. It is similar to conventional ionic liquids in terms of low vapor pressure and low flammability. In addition, they are biodegradable, non-toxic, inexpensive and reusable. Moreover, unlike ionic liquids, these solvents do not require a preliminary purification step. Their ability to serve as catalysts as well as solvents has also been explored in the field of synthetic organic chemistry [19–23].

It is well known that HFIP exhibits high hydrogen bonding donor ability, low nucleophilicity, high ionizing power and the ability to solvate water. Its effect on organic transformations [24–27] and notably the activation of hydrogen peroxide for oxidation reactions [28–30] are also well documented. Thus, as a part of our program for developing new DESs, we believe that HFIP is a good hydrogen-bond donor and can form deep eutectic mixture with choline chloride. Herein, we report the preparation of a new DES (ChCl/HFIP). Its catalytic activity and reusability toward the

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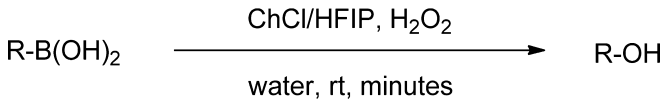


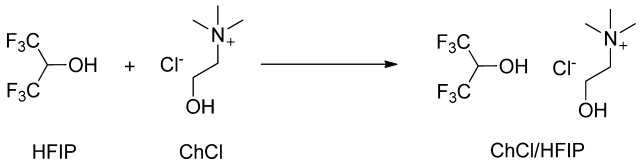
Scheme 1. ChCl/HFIP catalyzed the synthesis of phenols in water.

preparation of phenols from arylboronic acids is also investigated (Scheme 1).

2. Results and discussion

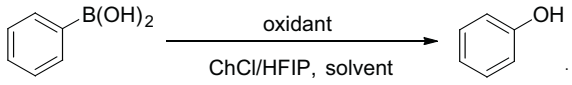
Firstly, the DES (ChCl/HFIP) was readily prepared by mixing the choline chloride with HFIP at 50 °C till a clear solution was obtained (Scheme 2). Several ratios of ChCl/HFIP were examined and 1:1.5 was selected as the best ratio, which was confirmed by NMR analysis. DSC analysis also clearly showed that the melting point of ChCl/HFIP was −37.2 °C. Moreover, ChCl/HFIP was very stable and could be easily stored on shelf without decomposition.

With this DES in hand, we started to optimize the reaction conditions using phenylboronic acid **1a** as the substrate (Table 1). When a mixture of **1a** (1 mmol) and aqueous H_2O_2 (2 mL, ca. 20 equiv) was stirred at room temperature for 1 h, only a trace amount of phenol **2a** was detected (Table 1, entry 1). When the reaction was carried out in other solvents such as acetonitrile,



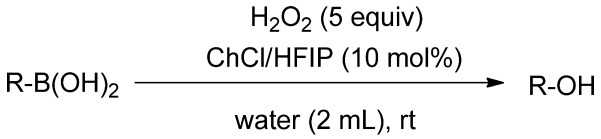
Scheme 2. Preparation of ChCl/HFIP.

Table 1
Optimization of reaction conditions.[a]



Entry	ChCl/HFIP (amount)	Oxidant (equiv)	Solvent	Time (min)	Yield[b] (%)
1	–	H_2O_2 (20)	–	60	Trace
2	–	H_2O_2 (20)	CH_3CN	60	<10
3	–	H_2O_2 (20)	EtOH	60	<10
4	–	H_2O_2 (20)	THF	60	<10
5	10 mol%	H_2O_2 (20)	CH_3CN	30	82
6	10 mol%	H_2O_2 (20)	EtOH	30	75
7	10 mol%	H_2O_2 (20)	THF	30	63
8	10 mol%	H_2O_2 (20)	–	5	87
9	10 mol%	H_2O_2 (10)	H_2O	5	94
10	10 mol%	H_2O_2 (5)	H_2O	5	95
11	10 mol%	H_2O_2 (2)	H_2O	5	65
12	5 mol%	H_2O_2 (5)	H_2O	5	70
13	20 mol%	H_2O_2 (5)	H_2O	5	87
14	20 mol%	H_2O_2 (5)	H_2O	5	82[c]
15	10 mol%	Oxone (5)	H_2O	60	Trace
16	10 mol%	TBHP (5)	H_2O	60	16
17	2 mL	H_2O_2 (2)	–	60	40
18	2 mL	H_2O_2 (5)	–	60	67
19	2 mL	H_2O_2 (10)	–	30	71
20	2 mL	H_2O_2 (20)	–	30	82

[a] *Reaction conditions*: phenylboronic acid (1 mmol), solvent (2 mL), oxidant, room temperature.
[b] Isolated yields.
[c] HFIP as catalyst.



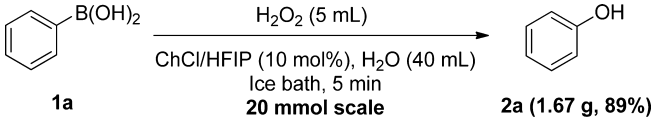
Scheme 3. Oxidative hydroxylation of arylboronic acid.

ethanol and tetrahydrofuran, the yield was only slightly increased. Surprisingly, the yields were greatly elevated in the presence of 10 mol% of ChCl/HFIP in both protic and aprotic solvents, although variations in yields were observed (Table 1, entries 5–7). When the reaction proceeded in the absence of any solvent, a 87% yield of **2a** was obtained (Table 1, entry 8). The influences of ChCl/HFIP and aqueous H_2O_2 were also evaluated, and the results clearly showed that the combination of 10 mol% of ChCl/HFIP and 5 equiv of aqueous H_2O_2 was the best choice, giving **2a** in 95% yield with 100% selectivity (Table 1, entry 10). When HFIP was used alone, more catalyst was required but the yield decreased (Table 1, entry 14). Other oxidants such as oxone and TBHP were also employed, however, poor yields were obtained after 1 h. Notably, increasing the amount of DES inhibited the reaction, which was attributed to a slow mass transfer that caused by the viscosity of the mixture (Table 1, entries 17–20) [19]. Although the viscosity was reduced by adding more aqueous H_2O_2, the reaction became violent and the selectivities decreased.

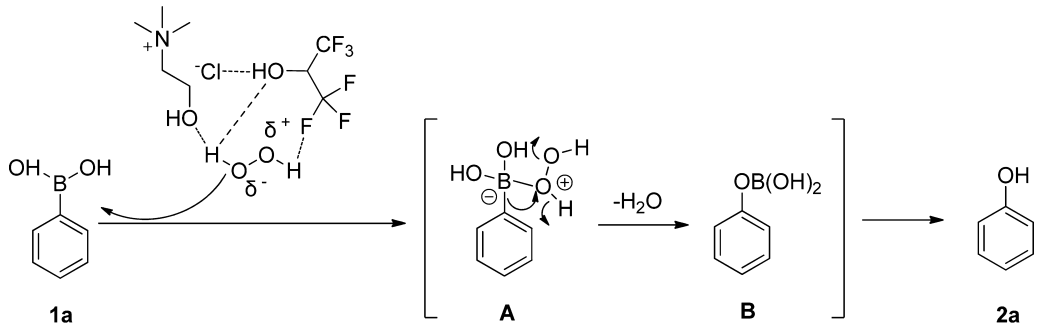
To evaluate the scope and limitations of the current procedure, a series of arylboronic acids were tested under the optimized reaction conditions from Table 1 (entry 10). The results were summarized in Scheme 3.

In general, arylboronic acids with either electron-withdrawing or electron-donating substituents such as halide, OCH_3, CF_3, CHO, NO_2, Ac, OBn underwent the reaction in good to excellent yields (80–95%). The conversion of electron-rich arylboronic acids was complete within 5 min, while relatively longer reaction time (30–60 min) was required for the electron-deficient arylboronic acids. It was worth noting that arylboronic acids bearing halides at para position provided the corresponding phenols **2d** and **2e** in excellent yields, as these compounds could be utilized for subsequent functionalization. Moreover, the oxidation-sensitive substituent such as **2g** tolerated the conditions and no overoxidation was observed. The steric effect was also negligible, giving the products **2k**–**2o** in good to excellent yields. Finally, the heteroarylboronic acids such as thiopheneboronic acid was examined and showed good compatibilities with the conditions (**2p**).

To further demonstrate the practicality and efficiency of the developed protocol, a scale-up reaction was performed using phenylboronic acid as substrate (2.44 g, 20 mmol). The reaction was carried out in ice bath with slowly addition of the hydrogen peroxide in order to avoid the over-oxidation of phenol. The solid product was precipitated in 89% yield under this condition and could be easily isolated by simple filtration. Moreover, one of the most important advantages employing DES as solvent or catalyst is the recyclability. Thus, a batch of reaction was performed in a 20 mmol scale to examine the recycling process. The recovery was very simple involving evaporation of the water after isolation of product by filtration. The deep eutectic mixture was reused without obvious loss in activity in five consecutive runs (89%, 89%, 88%, 88%, and 86% yield, respectively) (Scheme 4).



Scheme 4. Reusability of ChCl/HFIP.



Scheme 5. Plausible reaction mechanism.

As for the reaction mechanism, Berkessel et al. did a detailed mechanistic investigation of epoxidation of olefins by hydrogen peroxide in the presence of HFIP and multiple H-bond networks were believed to be the key factor [31]. Similarly, in this reaction, it was speculated that the DES could effectively activate the hydrogen peroxide through multiple H-bond networks. Later, the activated hydrogen peroxide attacked the boronic acid to generate intermediate **A**. Subsequent migration of the phenyl group from boron to oxygen atom of H_2O_2 generated boronate ester **B**, which was then hydrolyzed to give the final product phenol (Scheme 5) [32].

3. Conclusion

In summary, a rapid, green and practical oxidative hydroxylation of aryl/heteroaryl boronic acids was developed. The reactions proceeded at room temperature and provided the corresponding phenols in good to excellent yields with a few minutes. The DES (ChCl/HFIP) could be recovered by simple filtration and evaporation and reused for five runs without obvious loss of its activity. The broad substrate compatibility, metal- and additive-free conditions as well as gram-scale synthesis made this procedure more environmentally benign. Moreover, the formation of DES with ChCl also provided a new way to recover the HFIP.

4. Experimental

4.1. Method and apparatus

All reagents were obtained from local commercial suppliers and used without further purification. Melting points were determined with a WRS-1B apparatus and were uncorrected. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Advance 300 analyzer. All the products are known compounds and were identified by comparing of their physical and spectra data with those reported in the literature.

4.2. General procedure for preparation of ChCl/HFIP

Choline chloride (139.6 g, 100 mmol) and 1,1,1,3,3,3-hexafluoro-2-propanol (25.2 g, 150 mmol) were placed in a round bottom flask and stirred at 50 °C. After 3 h, a homogenous colorless liquid (164.8 g, 100%) formed, which was used directly for the reactions without purification.

4.3. General procedure for synthesis of compound **2**

A mixture of arylboronic acid (1.0 mmol), 30% H_2O_2 (5 equiv, 0.5 mL), water (2 mL) and ChCl/HFIP (10 mol%, 2 drops) was stirred at room temperature for the time indicated in Scheme 2. After completion of the reaction (indicated by TLC), the reaction mixture was extracted with EtOAc (3 × 10 mL). The organic layer was concentrated and the resulting crude products were purified by column chromatography on silica gel using PE/EtOAc as eluent to provide the desired products.

4.4. Gram-scale synthesis of **2a** and recovery of ChCl/HFIP

A reaction flask was charged with phenylboronic acid (2.44 g, 20 mmol), water (40 mL) and ChCl/HFIP (10 mol%, 40 drops), and the mixture was stirred in an ice bath. To this, 30% H_2O_2 (5 equiv, 5 mL) was added slowly. After addition of H_2O_2, the mixture became clear and the product was gradually precipitated at 0 °C. After filtration of the product, the water phase was further extracted with EtOAc (3 × 20 mL). The solid product was redissolved in organic layer, followed by concentration and purification by column chromatography. To the aqueous phase, a small amount of manganese dioxide was added to decompose the excess hydrogen peroxide. After filtration of the manganese dioxide, the catalyst ChCl/HFIP was readily recovered by evaporation of the water and reused for the next run.

4.4.1. Phenol (**2a**)

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33 (dd, J = 11.1, 4.2 Hz, 2H), 7.06 (d, J = 7.4 Hz, 1H), 7.00 (dd, J = 9.1, 7.9 Hz, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 155.1, 130.0, 121.0, 115.1.

4.4.2. p-Cresol (**2b**)

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.03 (d, J = 6.3 Hz, 2H), 6.73 (d, J = 6.3 Hz, 2H), 4.79 (s, 1H), 2.27 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.4, 130.3, 130.2, 115.3, 20.7.

4.4.3. 4-Methoxyphenol (**2c**)

^{1}H NMR (300 MHz, $CDCl_3$) δ 6.90–6.72 (m, 4H), 5.62 (s, 1H), 3.77 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.6, 149.6, 116.2, 115.0, 56.0.

4.4.4. 4-Chlorophenol (**2d**)

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.23–7.14 (m, 1H), 6.81–6.73 (m, 1H), 5.94 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 152.3, 132.2, 129.3, 121.9, 116.3, 110.3.

4.4.5. 4-Bromophenol (**2e**)

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37–7.28 (m, 2H), 6.77–6.67 (m, 2H), 5.51 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 154.7, 132.6, 117.3, 113.0.

4.4.6. 4-(Trifluoromethyl)phenol (**2f**)

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.51 (d, J = 8.4 Hz, 2H), 6.99–6.78 (m, 2H), 5.82 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.2, 127.4 (q, J_{C-F} = 3.8 Hz), 126.3, 123.2, 115.6.

*4.4.7. 4-Hydroxybenzaldehyde (**2g**)*

^{1}H NMR (300 MHz, $CDCl_3$) δ 9.85 (s, 1H), 7.93–7.70 (m, 2H), 7.11–6.91 (m, 2H), 6.80 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.5, 162.0, 132.7, 129.8, 116.2.

*4.4.8. 4-Nitrophenol (**2h**)*

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.32–8.08 (m, 2H), 7.07–6.85 (m, 2H), 6.71 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.8, 141.7, 126.4, 115.9.

*4.4.9. 1-(4-Hydroxyphenyl)ethanone (**2i**)*

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.96–7.85 (m, 2H), 7.70 (s, 1H), 6.99–6.88 (m, 2H), 2.56 (s, 3H); ^{13}C NMR (75 MHz, CDCl3) δ 198.6, 161.4, 131.2, 129.6, 115.7, 26.4.

*4.4.10. 4-(Benzyloxy)phenol (**2j**)*

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48–7.28 (m, 5H), 6.93–6.81 (m, 2H), 6.80–6.71 (m, 2H), 5.01 (s, 2H), 4.87 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.1, 149.8, 137.3, 128.7, 128.1, 127.6, 116.2, 116.0, 70.94 (s).

*4.4.11. 2,5-Dimethylphenol (**2k**)*

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.05 (d, J = 7.6 Hz, 1H), 6.72 (d, J = 7.6 Hz, 1H), 6.63 (s, 1H), 5.10–5.02 (m, 1H), 2.31 (s, 3H), 2.25 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.6, 137.2, 130.9, 121.6, 120.7, 115.8, 21.0, 15.4.

*4.4.12. 2-Bromophenol (**2l**)*

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 (dd, J = 8.0, 1.5 Hz, 1H), 7.23 (ddd, J = 8.2, 7.4, 1.5 Hz, 1H), 7.05 (dd, J = 8.2, 1.5 Hz, 1H), 6.82 (ddd, J = 8.0, 7.4, 1.5 Hz, 1H), 5.66 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 152.3, 132.2, 129.3, 121.9, 116.3, 110.3.

*4.4.13. Naphthalen-1-ol (**2m**)*

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.32–8.09 (m, 1H), 7.93–7.72 (m, 1H), 7.52 (ddd, J = 5.7, 4.2, 2.0 Hz, 2H), 7.46 (d, J = 8.4 Hz, 1H), 7.31 (dd, J = 14.1, 5.9 Hz, 1H), 6.82 (dd, J = 7.4, 0.9 Hz, 1H), 5.49 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 151.5, 134.9, 127.8, 126.6, 126.0, 125.4, 124.5, 121.7, 120.8, 108.7.

*4.4.14. Naphthalen-2-ol (**2n**)*

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.78 (t, J = 7.7 Hz, 2H), 7.68 (d, J = 8.2 Hz, 1H), 7.45 (ddd, J = 8.2, 6.9, 1.3 Hz, 1H), 7.35 (ddd, J = 8.0, 6.9, 1.2 Hz, 1H), 7.16 (d, J = 2.4 Hz, 1H), 7.14 (dt, J = 8.7, 2.5 Hz, 2H), 7.12 (dd, J = 8.7, 2.5 Hz, 1H), 5.45 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.4, 134.7, 130.0, 129.0, 127.9, 126.6, 123.8, 117.9, 109.7.

*4.4.15. 1,1′-Biphenyl]-2-ol (**2o**)*

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.53–7.46 (m, 4H), 7.46–7.37 (m, 1H), 7.29 (dd, J = 12.1, 4.6 Hz, 2H), 7.03 (dd, J = 11.4, 4.2 Hz, 2H), 5.29 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 152.5, 137.2, 130.4, 129.5, 129.4, 129.3, 128.3, 128.0, 121.0, 116.0.

*4.4.16. Thiophen-2-ol (**2p**)*

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.58 (td, J = 4.32, 2.04 Hz, 1H), 6.41 (td, J = 4.42, 1.42 Hz, 1H), 4.14 (t, J = 1.76 Hz, 2H).

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