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(*S*)-2-Aryl-4,4-diphenyl-3,1,2-oxazaboro[3.3.0]octanes: Efficient catalysts for the asymmetric borane reduction of electron-deficient ketones

Han Liu, J.X. Xu *

Key Laboratory of Bioorganic Chemistry and Molecular Engineering of Ministry of Education, College of Chemistry and Molecular Engineering, Peking University, Beijing 100871, PR China

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Abstract

The obvious influence of electronic effects of ketones on the enantioselectivities was observed previously in the oxazaborolidine-catalyzed asymmetric borane reduction of ketones. On the basis of the catalytic reduction mechanism, the electronic effect of organocatalysts, B-aryl-substituted oxazaborolidines, was tuned rationally to improve the enantioselectivities of the electron-deficient ketones in the reduction. The results indicate that all B-aryloxazaborolidines show excellent enantioselectivities for the electron-deficient ketones. This indicates that B-aryloxazaborolidines show better enantioselectivities than B-unsubstituted and B-methoxy-substituted oxazaborolidines for the electron-deficient ketones.

Keywords: Asymmetric reduction; Borane; Electronic effect; Enantioselectivity; Ketone

1. Introduction

Chiral 1,3,2-oxazaborolidine-catalyzed borane reduction of prochiral ketones to chiral secondary alcohols is one of the most important reactions in asymmetric syntheses, which has been widely used during the past decade [1]. Numerous new efficient oxazaborolidine catalysts have been reported and a plethora of applications have appeared till now. In comparison with the numerous attempts to search for new catalysts to improve the enantioselectivity, several papers have concentrated on the mechanistic investigation of the catalytic asymmetric reduction [2,3]. Some papers have been paid attention to the factors which affect the enantioselectivity in the asymmetric reduction, such as the structure [1,2,4], the stability [2a,5] (including dimerization) and the loading amount [2a,5a,6] of the catalyst, the borane source [7] and amount [2a,6c], the order and rate of the addition of a ketone or borane into a reductive system [1d,6c], the reduction temperature [5d,6c,8], the solvent [5a,6c,7c], the additive [8g,9,10], the secondary reduction [9a,11], the stabilizer in borane [12], the electronic effects of ketones [4a,5a,10,13], etc. Although a few papers have considered on the influence of the electronic effects of catalysts on the enantioselectivity, most of the results indicated that no obvious influence has been observed in the asymmetric reduction [4a,5a]. Very recently we investigated the influence of the electronic effects of ketones on the enantioselectivity in the asymmetric reduction and found that the electron-deficient ketones generally give lower enantioselectivities [10].

The factors governing enantioselectivity in the catalytic asymmetric reaction are usually interpreted in steric terms [14] affected by the temperature and solvent, etc. [5a,6c,7c,8]. Electronic effects have become an important factor to control the enantioselectivity in recent investigations [15]. It should be very useful to understand all factors, which affect the enantioselectivity, and it will be helpful to apply the asymmetric reaction effectively to a variety of substrates. Herein, we wish to present our investigation on the influence of the electronic effects of catalysts on the enantioselectivity in the asymmetric borane reduction of the electron-deficient ketones.

2. Experimental

2.1. General methods

^{1}H spectra were recorded on a Varian Mercury 200 (200 MHz) and Mercury Plus 300 (300 MHz) spectrometer in

* Corresponding author. Tel.: +86 10 6275 1497; fax: +86 10 6275 1708.
E-mail address: jxxu@pku.edu.cn (J.X. Xu).

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$CDCl_3$ solution with TMS as an internal standard and chemical shifts are reported in ppm. Optical rotations were measured on a Perkin-Elmer Model 341LC polarimeter with a thermally jacketed 10 cm cell (concentration *c* given as g/100 mL). HPLC analyses were performed on an HP1100 HPLC equipment. The e.e. values were determined by HPLC analysis with chiralcel AD, AS, OB, OD, or OD-H columns (4.6 mm × 250 mm) with a mixture of *n*-hexane–isopropanol as an eluent. Borane–dimethyl sulfide complex and substituted arylboric acids were purchased from Acros Chemicals Co. Toluene was heated under reflux over sodium and distilled prior to use.

2.2. General procedure for the preparation of catalysts *1d–g*

A 25 mL round-bottomed flask equipped with a stirring bar and a 10 mL pressure-equalizing addition funnel (containing a cotton plug and ca. 10 g of 5 Å molecular sieves, and functioning as a Soxhlet extractor) [16]. A mixture of (*S*)-diphenylprolinol (0.05 mmol, 12.7 mg) and arylboronic acid (0.05 mmol) was solved in 15 mL of dry toluene. The resulting solution was heated to reflux for 12 h. Then most of the solvent was distilled off and the residue (ca. 3 mL) was cooled to room temperature. The addition funnel was removed off and the flask was airproofed quickly to avoid moisture. The catalyst can be used directly without further purification.

2.3. General procedure for the asymmetric reduction of ketones using catalysts *1d–g*

To a solution of the catalyst (0.05 mmol, 10 mol%) that was freshly prepared in dry toluene, 2 mol/L borane–dimethyl sulfide complex in THF (0.25 mL, 0.5 mmol) was added under a nitrogen atmosphere at room temperature. Then the solution was warmed or cooled to the desired temperature and stirred for 15 min. A solution of ketone (0.5 mmol) in 4 mL of toluene was then added dropwise over 1 h. After the addition, the resulting solution was stirred for 4 h and then quenched with 0.5 mL of methanol in an ice bath. After concentration under reduced pressure, the residue was purified on a silica gel column with a mixture of petroleum ether (60–90 °C) and ethyl acetate (5:1, v/v) as an eluent to give chiral secondary alcohol as a colorless oil. The spectral and analytical data of all obtained alcohols are in agreement with those reported in the literature [10,17,18]. The e.e. value was determined by chiral HPLC analysis.

2.4. (*R*)-1-(4-Bromo-3-nitrophenyl)ethanol (*3c*)

Colorless liquid; $[\alpha]_D^{20}$ = +25.0 (c, 0.5, CH_2Cl_2), e.e. 98%; ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.50 (d, J = 6.4 Hz, 3H, CH_3), 1.88 (s, br, 1H, OH), 4.95 (q, J = 6.4 Hz, 1H, CH), 7.43 (d, J = 8.4 Hz, 1H, ArH), 7.69 (d, J = 8.4 Hz, 1H, ArH), 7.86 (s, 1H, ArH). ^{13}C NMR (50 MHz, $CDCl_3$) δ: 25.38, 68.79, 112.74, 122.53, 130.13, 135.00, 146.57, 147.01. MS (EI) *m/z*: 245 (M^+), 247, 230, 232, 185, 187, 155, 157, 102, 75, 51; IR ν (cm^{-1}): 3350 (OH), 2975, 2925, 1534, 1355. Racemate was reported in the literature [17].

2.5. (*R*)-1-(3,5-dinitrophenyl)ethanol (*3d*)

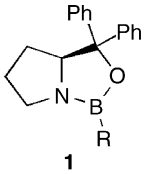
Colorless solid; $[\alpha]_D^{20}$ = +25.9 (c, 1.0, $CHCl_3$), e.e. 98%; ^{1}H NMR (200 MHz, $CDCl_3$) δ: 1.61 (d, J = 6.4 Hz, 3H, CH_3), 2.09 (s, br, 1H, OH), 5.16 (q, J = 6.4 Hz, 1H, CH), 8.60 (s, 2H, ArH), 8.95 (s, 1H, ArH); ^{13}C NMR (50 MHz, $CDCl_3$) δ: 25.67, 68.69, 116.01, 117.65, 125.75, 148.61; MS (EI) *m/z*: 211 ($M^+ - 1$), 197, 166, 152, 105, 91, 75, 51; IR ν (cm^{-1}): 3353 (OH), 2916, 1541, 1345, 731. Racemate was reported in the literature [18].

3. Results and discussion

It has been proven that chiral (*S*)-2-substituted 4,4-diphenyl-3,1,2-oxazaboro[3.3.0]octanes **1**, derived from (*S*)-2-(diphenylhydroxymethyl)pyrrolidine, with several different substituents (such as R is H for **1a**, Me for **1b**, MeO for **1c** and Ph for **1d**, as representatives, 4-FPh for **1e**, 4-ClPh for **1f**, 3-NO_2Ph for **1g**, Scheme 1) on B atom are the most effective catalysts in the asymmetric reduction with excellent yields and enantioselectivities for a wide variety of ketones [1,2,6]. We have found that catalyst **1c** is an effective, convenient and practical catalyst because it can be prepared from (*S*)-2-(diphenylhydroxymethyl)pyrrolidine and inexpensive trimethyl borate in situ and used directly without any further separation and purification [6]. However, after the investigation on the influence of the electronic effects of ketones on the enantioselectivity [10] it seems that catalyst **1c** is not efficient to the ketones with electron-withdrawing groups. The results prompt us to consider tuning the influence of the electronic effects of the catalysts rationally to improve the enantioselectivity of the reduction of the ketones with electron-withdrawing groups on the basis of the reaction mechanism of the asymmetric reaction.

On the basis of our previous investigation and analysis [10] the coordination step in the catalytic cycle is a key step for the enantioselectivity, in other words, an e.e.-determining step in the reduction cycle. The efficient coordination between a catalyst and a ketone will afford the excellent enantioselectivity. To improve the enantioselectivity of the electron-deficient ketones, which have a relative hard oxygen atom (hard base), according to the Pearson's hard–soft acid–base rule, we need to use a catalyst with a relative hard boron atom (hard acid), e.g. the catalyst has an electron-withdrawing group on its boron atom (Scheme 2).

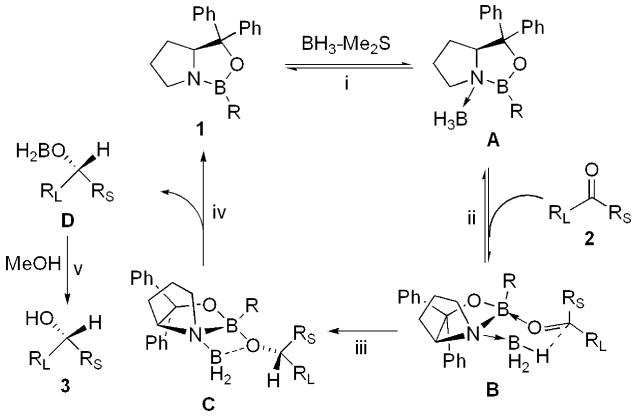
We hope to tune the electronic effects of the catalysts by using (*S*)-2-aryl-4,4-diphenyl-3,1,2-oxazaboro[3.3.0]octanes **1d–g**



1

a: R = H, **b:** R = Me, **c:** R = MeO, **d:** R = Ph,
e: R = 4-FPh, **f:** R = 4-ClPh, **g:** R = 3-NO_2Ph

Scheme 1. (*S*)-2-Substituted 4,4-diphenyl-3,1,2-oxazaboro[3.3.0]octane catalysts.



Scheme 2. General accepted mechanism for the oxazoborolidine-catalyzed asymmetric borane reduction of ketones.

and to improve the enantioselectivity of the electron-deficient ketones in the asymmetric borane reduction. Although the influence of the electronic effect on the enantioselectivity in the asymmetric borane reduction of ketones has been considered several times in literatures [4a,5a,13] no obvious influence has been observed when the B-alkyl and aryl-substituted oxazaborolidines **1b** and **1d** were used as the catalysts, and different alkyl *para*-substituted aryl ketones were used as substrates except for the reports of Xu et al. [10] and Corey and Helal [13c]. Mathre et al. investigated the influence of *para*-substituted acetophenones using the oxazaborolidine **1b** as the catalyst under stoichiometric reduction conditions [5a]. Jones et al. paid much attention to the influence using the oxazaborolidine **1b** itself and its derivatives with different *para*-substituents on the phenyl ring of the diphenylprolinol moiety, the oxazaborolidine **1d** itself and its derivatives with different *para*-substituents on the phenyl ring attached to the boron atom in the asymmetric reduction in dichloromethane [4a]. Unfortunately, no significant electronic effect was observed in all of their cases. However, Xu et al. [10] and Corey and Helal 13c] did observe obvious influence of the electronic effects of ketones on the enantioselectivity in the asymmetric reduction, respectively.

To further investigate the influence and to hope to improve the enantioselectivity via tuning the electronic effect of catalysts rationally, a series of (*S*)-2-aryl-4,4-diphenyl-3,1,2-oxazaboro[3.3.0]octanes were prepared. To optimize the reduction temperature, the enantioselectivities of a model substrate *para*-nitroacetophenone at different temperatures were first determined for all B-aryl catalysts **1d–g** (Fig. 1).

The results indicate that all B-aryl catalysts **1d–g** show excellent enantioselectivities at 25 °C possibly because aryl groups are more electron-withdrawing groups than H, alkyl and alkoxy groups according to their Hammett constants. To observe the influence of the electronic effects on the enantioselectivity, the two electron-deficient ketones, *para*-nitroacetophenone and propiophenone were reduced under the catalysis of B-aryl catalysts **1d–g** at 0 and 25 °C, respectively. In our asymmetric reduction, the catalysts were prepared firstly via the azeotropic distillation dehydrolysis from arylboric acids and (*S*)-

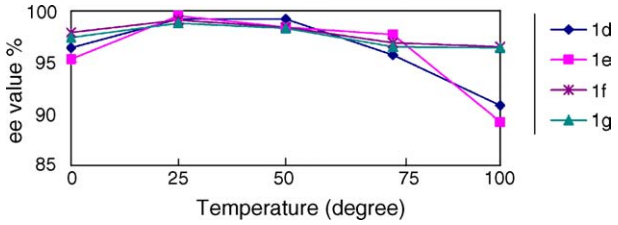
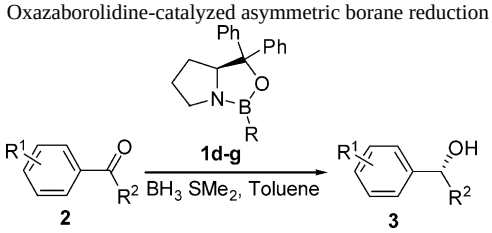


Fig. 1. Enantioselectivities of the reduction of *para*-nitroacetophenone catalyzed by catalysts **1d–g** at different temperatures.

2-(diphenylhydroxymethyl)pyrrolidine in toluene according to the literature [16]. After addition of borane, a ketone was added dropwise at 0 or 25 °C during a period of 1 h. After stirring for 4 h, the reaction mixture was quenched with methanol and usual workup, enantiomeric excess value was determined using a chiral column on HPLC. The results are presented in Table 1. Both of the two ketones give excellent enantioselectivities and yields. *Para*-nitropropiophenone affords relative lower enantioselectivities than *para*-nitroacetophenone due to the increase from methyl to ethyl. The enantioselectivities seem to increase with increasing the electron-withdrawing group of the aryl attached on the boron atom in the catalysts. However, no obvious electronic effect on the enantioselectivity was observed because all of the enantioselectivities are too high. To further verify the influence, two more electron-deficient acetophenones, 4-bromo-3-nitro-acetophenone and 3,5-dinitroacetophenone were designed and prepared [19]. Both of them give excellent enantioselectivities without obvious differences. Although no obvious influence of the electronic effects of catalysts on the enantioselectivity was observed in the asymmetric borane reduction of the electron-deficient ketones, the results indicate that (*S*)-2-aryl-4,4-diphenyl-3,1,2-oxazaboro[3.3.0]octanes are efficient catalysts for the asymmetric borane reduction of the electron-deficient ketones. Two series of electron-efficient ketones, *para*-haloacetophenones **2e–g** and propiophenones **2h–j** were also asymmetrically reduced under the same condition. All of them gave excellent enantioselectivities. To further support our conclusion, two electron-donating ketones, *p*-methoxyacetophenone **2k** and *p*-methylthiopropiophenone **2l** were also asymmetrically reduced under the same condition. Both of them gave excellent enantioselectivities. The results are summarized in Table 1.

The electronic tuning of asymmetric catalysts can change the stereoselectivity in catalytic asymmetric reactions [15a,b,d]. Regulation of electronic effects of catalysts is another important pathway to find effective catalysts. Regulating the electronic effect match of catalysts and substrates should be important especially for the asymmetric transformation where stereoselectivity relies purely on nonbond interaction. The interaction performs the stereochemical communication between a chiral catalyst and a substrate. The regulation should be one of very useful methods to improve enantioselectivity in an asymmetrically catalytic reaction. The present investigation indicates that the electronic tuning of the catalysts can improve their enantioselectivity in the asymmetric borane reduction.

Table 1
Oxazaborolidine-catalyzed asymmetric borane reduction of electron-deficient ketones



Entry	Ketone	R^1	R^2	Temperature (°C)	Catalyst **1d**		Catalyst **1e**		Catalyst **1f**		Catalyst **1g**	
					Yield (%)[a]	e.e. (%)[b]	Yield (%)[a]	e.e. (%)[b]	Yield (%)[a]	e.e. (%)[b]	Yield (%)[a]	e.e. (%)[b]
1	**2a**	NO_2	Me	0	90	96[c]	94	95	99	98	99	97
2	**2a**	NO_2	Me	25	95	99	90	99.5	99	91	92	99
3	**2b**	NO_2	Et	0	84	95[d]	85	96	92	95	89	93
4	**2b**	NO_2	Et	25	86	91	78	92	88	92	94	91
5	**2c**	4-Br-3-NO_2	Me	0	92	96[e]	92	95	87	96	98	99.5
6	**2c**	4-Br-3-NO_2	Me	25	98	98						
7	**2d**	3,5-$(NO_2)_2$	Me	25	100	98[f]	87	95	100	96	89	97
8	**2e**	Br	Me	25	85	98[g]						
9	**2f**	Cl	Me	25	80	98[h]						
10	**2g**	F	Me	25	71	96[i]						
11	**2h**	Br	Et	25	91	93[j]						
12	**2i**	Cl	Et	25	94	91[g]						
13	**2j**	F	Et	25	61	95[i]						
14	**2k**	MeO	Me	25	97	98[k]						
15	**2l**	MeS	Et	25	90	>99[l]						

[a] Isolated yields after the column chromatography.
[b] e.e. values were determined by HPLC analyses using AS, OD, OD-H, OB, or AD chiral columns (4.6 mm × 250 mm, Chiralcel) and a mixture of *n*-hexane and 2-propanol as an eluent. Configuration was assigned according to the rotation value. In each case a positive rotation was obtained, indicating that the selectivity was for the (*R*)-enantiomer in agreement with reported work (ref. [10]).
[c] AS column, *n*-hexane:2-propanol (95:5, v/v), 0.8 mL/min, 254 nm.
[d] AD column, *n*-hexane:2-propanol (97:3, v/v), 0.8 mL/min, 254 nm.
[e] AD column, *n*-hexane:2-propanol (97:3, v/v), 1.0 mL/min, 254 nm. Configuration was assigned on the basis of the reaction mechanism.
[f] AD column, *n*-hexane:2-propanol (95:5, v/v), 1.0 mL/min, 254 nm. Configuration was assigned on the basis of the reaction mechanism.
[g] OD-H column, *n*-hexane:2-propanol (97:3, v/v), 0.8 mL/min, 220 nm.
[h] OD column, *n*-hexane:2-propanol (97:3, v/v), 0.8 mL/min, 220 nm.
[i] OB column, *n*-hexane:2-propanol (99:1, v/v), 0.8 mL/min, 220 nm.
[j] OB column, *n*-hexane:2-propanol (97:3, v/v), 0.8 mL/min, 220 nm.
[k] OB column, *n*-hexane:2-propanol (95:5, v/v), 0.8 mL/min, 220 nm.
[l] OB column, *n*-hexane:2-propanol (95:5, v/v), 1.0 mL/min, 220 nm.

4. Conclusions

On the basis of the catalytic mechanism of the oxazaborolidine-catalyzed asymmetric borane reduction of ketones, the electronic effect of organocatalysts, B-aryl-substituted oxazaborolidines was rationally tuned to improve the enantioselectivity of the electron-deficient ketones in the reduction. The results indicate that all B-aryloxazaborolidines show the excellent enantioselectivity for the electron-deficient ketones. It has also been found that B-aryloxazaborolidines show much better enantioselectivity than B-unsubstituted and B-methoxy-substituted oxazaborolidines for the electron-deficient ketones.

On the basis of the investigation, one can select a suitable catalyst for borane asymmetric reduction of ketones with a variety of functional groups. We hope that the present study has addressed an important issue regarding the catalyst selection in the oxazaborolidine-catalyzed asymmetric reduction of ketones. To achieve excellent enantioselectivity, we recommend the oxazaborolidine **1c** as the most suitable catalyst for the ketones with electron-donating groups because of its convenient preparation, economic starting materials and high efficiency, the oxazaborolidine **1d** as a good choice for the ketones with electron-withdrawing groups.

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