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The preparation of optically active boronic ester substituted Δ^2-isoxazolines

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Abstract

In this paper we report our recent results in the area of nitrile oxide cycloaddition to optically active vinylboronic esters to afford optically active boronic ester substituted Δ^2-isoxazolines. In these studies, a number of optically active diols were investigated and TADDOLs have been found to afford the best diastereoselectivity. The mixture of diastereomers obtained in these reactions can be readily purified by formation of the diethanolamine–boron complexes and recrystallized to afford the pure enantiomers. © 1999 Elsevier Science S.A. All rights reserved.

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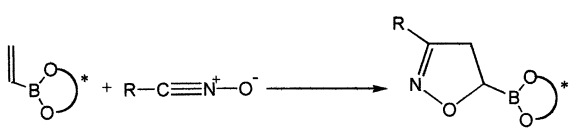
1. Introduction

The Δ^2-isoxazolines produced by the 1,3-dipolar cycloaddition of a nitrile oxide to an alkene have proven to be extremely useful compounds in organic chemistry [1a,b,c,d]. Among the various classes of compounds which have been prepared from these cycloadducts are enones [1a], 1,3 amino-alcohols [1e], and β-hydroxy ketones [1a]. The boronic ester functionality has also enjoyed considerable use in organic synthesis [2a,b]. Procedures exist for the direct conversion of boronic esters into alcohols [2b], aldehydes [2b,c], carboxylic acids [2b,c], and amines [2b,c]. It has also been shown that it is possible to homologate boronic esters by inserting a methylene into the carbon–boron bond [3a,b,c]. The ability to carry out the homologation of a boronic ester coupled with the large number of functional groups which it represents has resulted in the boronic ester becoming an extremely versatile functional group. We report in this paper our recent results which combine the usefulness of the Δ^2-isoxazoline with that of the boronic ester, by carrying out nitrile oxide cycloadditions with optically active vinylboronic esters to afford optically active boronic ester substituted Δ^2-isoxazolines [4].

We have shown previously that vinylboronic esters are very reactive dipolarophiles in nitrile oxide cycloadditions [5a,b] and the resulting boronic ester containing Δ^2-isoxazolines can be transformed into a variety of useful products [6]. Our goal in this project was to extend this methodology to allow for the preparation of optically active boronic ester containing Δ^2-isoxazolines. There are several methods by which this could, be undertaken. One approach involves the use of an optically active diol as a chiral auxiliary on the boronic ester functional group. Nitrile oxide cycloaddition to the vinylboronic ester would afford the Δ^2-isoxazoline as a mixture of diastereomers (Eq. (1)). Another option would be to place the chiral auxiliary elsewhere in the vinylboronic ester. We recently reported the use of a camphorsultam substituted vinylboronic ester in this approach [7a,b].



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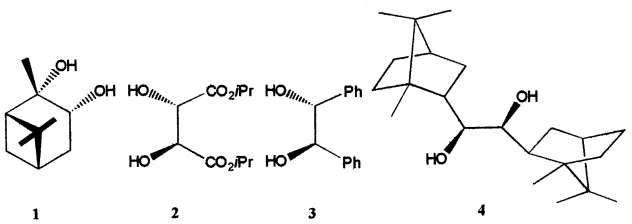
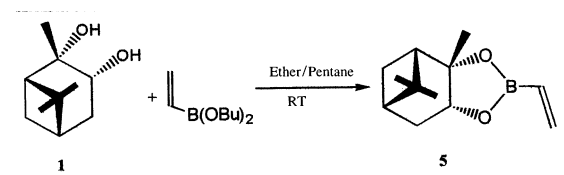
The route depicted in Eq. (1) seems particularly attractive in light of the elegant work of Matteson and coworkers employing chiral diols as ligands on boronic esters [2a] and because of the large number of optically active diols available. Our initial efforts in this project were directed at exploring optically active 1,2-diols as chiral auxiliaries in this reaction. Some of the optically active 1,2-diols explored in this reaction include pinanediol (**1**) [2a], di-isopropyl tartrate (**2**), dihydrobenzoin (**3**) and a diol prepared in our laboratories [8] for use as a ligand for titanium catalysts (**4**).



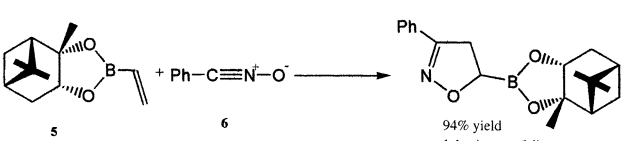
1 2 3 4

Preparation of the optically active vinylboronic esters was readily accomplished by treatment of the 1,2-diol with dibutyl vinylboronate [9] in an ether/pentane mixture at room temperature. An example of this transesterification employing pinanediol (**1**) is shown in Eq. (2).



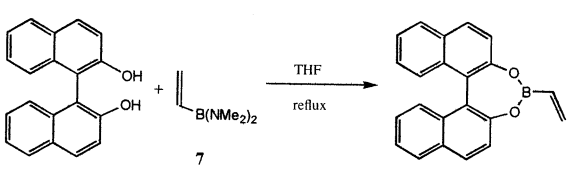
1 5 (2)

Nitrile oxide cycloaddition of benzonitrile oxide (**6**) with (**5**) afforded the Δ^2-isoxazoline in 94% yield as a 1:1 mixture of diastereomers (Eq. (3)). All other optically active 1,2-diols explored in this reaction also afforded the cycloadduct in high yield but gave very low levels of diastereoselectivity. Based on the low levels of diastereoselectivity obtained with 1,2-diols we decided to explore the use of 1,4-diols as chiral auxiliaries in this reaction.



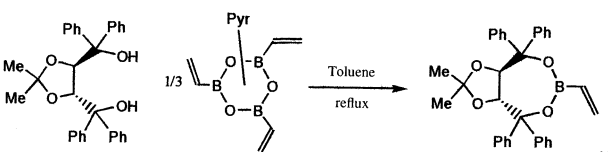
5 6 94% yield 1:1 mixture of diastereomers (3)

Binaphthol [10] and TADDOLs [11a,b] have been shown to be very effective chiral auxiliaries in a variety of reactions. Attempts to prepare the binaphthol substituted vinylboronic ester by reaction of binaphthol with dibutyl vinylboronate [9] or vinylboronic anhydride–pyridine complex [12] proved to be troublesome and an alternative method was explored. Reaction of bis-(dimethylamino)vinyl borane (**7**) and binaphthol in THF afforded the binaphthol substituted optically active vinylboronic ester (**8**) (Eq. (4)). Nitrile oxide cycloaddition of benzonitrile oxide (**6**) with (**8**) afforded the Δ^2-isoxazoline as a 2:1 mixture of diastereomers. Reactions employing vinylboronic ester (**8**) proved difficult to carry out due to the inherent instability of this compound towards hydrolysis of the binaphthol unit. It also proved difficult to determine the diastereoselectivity of this reaction from the crude reaction mixture due to the tendency of the binaphthol ligand to undergo hydrolysis during the work-up procedure. In an effort to develop a reliable method for the determination of the diastereoselectivity in this reaction the exchange of binaphthol with pinanediol (**1**) was explored. This exchange reaction proved to be extremely rapid and complete replacement of the binaphthol with pinanediol was readily accomplished. The ratio of diastereomers could be easily determined by examination of the ^{1}H-NMR spectra of the pinanediol containing boronic ester substituted Δ^2-isoxazolines. This exchange afforded a quick, easy, and reliable method for the determination of the diastereoselectivity in these cycloadditions. Although binaphthol proved to be a more efficient chiral auxiliary than the 1,2-diols studied, the moisture sensitivity of **7** and **8** along with the difficulty associated with their preparation made the exploration of other chiral auxiliaries attractive.



7 8 (4)

TADDOLs have been shown to be very effective chiral auxiliaries in a wide variety of organic reactions [11a,b]. The preparation of the TADDOL substituted vinylboronic ester (**9**) was easily accomplished by reaction of the TADDOL with vinylboronic anhydride–pyridine complex [12] in toluene at reflux (Eq. (5)).

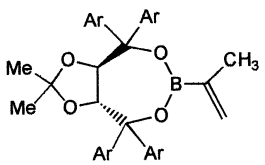


9 (5)

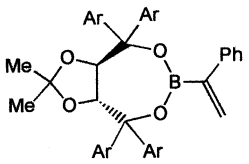
Nitrile oxide cycloaddition of benzonitrile oxide (**6**) with (**9**) proceeded smoothly to afford the cycloadduct as a 2:1 mixture of diastereomers. It again proved difficult to accurately determine the diastereoselectivity on the crude reaction mixture owing to the tendency of the TADDOL to undergo hydrolysis during work-up. Treatment of the crude cycloaddition mixture with pinanediol afforded the pinanediol containing boronic ester substituted Δ^2-isoxazoline. The determination of

the diastereoselectivity of the cycloaddition reaction was easily carried out by ^{1}H-NMR analysis of the crude exchange reaction mixture. A variety of TADDOLs (**10a**–**e**) [11a] were explored in this reaction and the selectivities obtained are shown Table 1 below. Attempts to increase the selectivity of these cycloadditions by changing solvent or temperature of the reaction did not have any significant impact on the selectivity.

In connection with a synthetic project being carried out in our laboratories, we wanted to explore the use of α-substituted optically active vinylboronic esters in this reaction. Two of the α-substituted vinylboronic esters explored in this reaction were **11** and **12**.



11, Ar=3,5-(Me)$_2$Ph

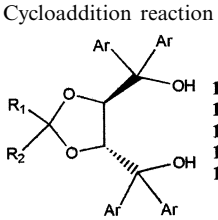


12, Ar=3,5-(Me)$_2$Ph

When the reaction of benzonitrile oxide (**6**) was carried out with **11** and **12** we were delighted to find much higher levels of asymmetric induction than were observed with the unsubstituted vinylboronic esters. The selectivities in these reactions were also determined by exchange of the TADDOL with pinanediol and ^{1}H-NMR analysis. Vinylboronic ester **11** afforded an 8:1 mixture of diastereomers and boronic ester **12** gave a 5:1 mixture of diastereomers. The increased level of asymmetric induction displayed by **11** and **12** is currently under further investigation.

Separation of the mixture of diastereomers obtained from these cycloadditions proved to be difficult by normal methods. At this point we decided to explore the possibility of carrying out the cycloaddition, exchanging the TADDOL for diethanolamine, and recrystallizing the diethanolamine–boron complexes as a means of separating the isomers obtained in the reaction [2b]. This allowed purification of the mixture of isomers obtained in the cycloaddition reaction. Replacement of the diethanolamine with pinanediol confirmed that the pure enantiomers could be isolated. The TADDOL could also be readily recovered in $>90\%$ yield and reused. Further studies are currently underway in our laboratories including the application of the these optically active boronic ester substituted Δ^2-isoxazolines to the synthesis of natural products and biologically important molecules.

Table 1
Cycloaddition reaction selectivities of TADDOLs



10a R_1, R_2=Me, Ar=3,5-(Me)$_2$Ph
10b R_1, R_2=Me, Ar=1-Napthyl
10c R_1, R_2=Me, Ar=2-Napthyl
10d R_1=H, R_2=Ph, Ar=3,5-(Me)$_2$Ph
10e R_1=Me, R_2=Ph, Ar=3,5-(Me)$_2$Ph

TADDOL	Nitrile oxide	Ratio of diastereomers
10a	PhCNO	2.8:1
10b	PhCNO	3:1
10b	*t*-BuCNO	2.5:1
10c	PhCNO	2.2:1
10d	PhCNO	2.5:1
10e	PhCNO	2.8:1

2. Experimental

Unless otherwise noted, materials were obtained from commercial suppliers and were used without further purification. Trimethyl borate and tri-isopropyl borate were freshly distilled prior to use. Tetrahydrofuran (THF) and diethyl ether were freshly distilled from sodium/benzophenone ketyl under nitrogen. High resolution mass spectra (HRMS) were obtained on a AG Autospec GC/MS spectrometer. ^{1}H-NMR spectra were measured in $CDCl_3$ on a Bruker AM 360 spectrometer (360 MHz) or a Hitachi RS-1200 (60 MHz). ^{13}C-NMR spectra were recorded in $CDCl_3$ at 90.0 MHz (Bruker AM 360), unless otherwise specified. ^{11}B-NMR spectra were measured in $CDCl_3$ on a Bruker AM 360 spectrometer and $BF_3{\cdot}Et_2O$ was used as a reference.

2.1. Pinanediol vinylboronic ester (*5*)

A solution of (+)-pinanediol (518 mg, 3.04 mmol) and dibutyl vinylboronic ester (560 mg, 3.04 mmol) in pentane/ether (10:1) (10 ml) was stirred at room temperature (r.t.) for 6 h. The solvent was removed under reduced pressure and the residue was purified by Kugelrohr distillation (61°C, 0.1 mmHg) to afford the product as a clear oil (604 mg, 98%). ^{1}H-NMR (360 Hz, $CDCl_3$) δ 6.15 (dd, $J=19.4$, 4.3 Hz, 1H), 6.04 (dd, $J=13.6$, 4.3 Hz, 1H), 5.90 (dd, $J=19.4$, 13.6 Hz, 1H), 4.31 (dd, $J=8.9$, 1.9 Hz, 1H), 2.37–2.33 (m, 1H), 2.27–2.21 (m, 1H), 2.15–2.10 (m, 1H), 1.95–1.90 (m, 2H), 1.41(s, 3H), 1.31 (s, 3H), 1.15 (d, $J=10.9$ Hz, 1H), 0.88 (s, 3H).

2.2. Cycloaddition product from reaction of *5* with *6*

A solution of pinanediol vinylboronic ester (**5**) (418 mg, 2.03 mmol) and phenylhydroximic acid chloride in THF (20 ml) was cooled to −78°C. Et_3N (207 mg, 2.05 mmol) in THF (5 ml) was added dropwise to the mixture at −78°C. After 12 h, the reaction was allowed to warm to r.t. The triethylamine hydrochloride salt was removed by filtration and the filtrate was

concentrated under reduced pressure. The residue was purified by Kugelrohr distillation (135°C, 0.05 mmHg) to afford a pale yellow oil (94%). IR (neat) 3005, 2980, 1570, 1450 cm^{-1}. ^{1}H-NMR (360 MHz, $CDCl_3$) δ 7.75–7.68 (m, 2H), 7.42–7.37 (m, 3H), 4.42–4.38 (m, 1H), 3.54–3.50 (m, 1H), 3.38–3.30 (m, 1H), 2.38–2.35 (m, 1H), 2.27–2.24 (m, 1H), 2.13–2.09 (m, 1H), 1.93–1.90 (m, 1H), 1.45 (s, 3H), 1.30 (s, 3H), 1.17–1.13 (m, 1H), 0.86 (s, 3H). ^{13}C-NMR (90 MHz, $CDCl_3$) 156.5, 129.9, 129.6, 128.5, 126.8, 86.9, 78.6, 68.1, 51.8, 39.3, 38.2, 38.1, 35.0'28.4, 26.9, 26.3, 23.8. MS *m/z* 55 (79), 67 (56), 77 (100), 104 (79), 137 (59), 153 (58), 174 (57), 325 (29). HRMS Calc. for $Cl_{19}H_{24}BNO_3$, 325.184. Found 325.184.

2.3. Vinylboronic anhydride–pyridine complex

A stirred suspension of dibutyl vinylboronate (15.0 g, 82.0 mmol), phenothiazine (0.80 g, 0.40 mmol) and water (40 ml) was distilled (ca. 55°C, 20 mmHg) through a 30 cm vigreux column until the butanol/water azeotrope and the majority of the water was removed to leave a moist residue. The residue of ethyleneboronic acid was treated with pyridine (30 ml) and stirred for 12 h. The water/pyridine azeotrope was distilled off (ca. 55°C, 20 mmHg) by a similar procedure above and the residue was purified by Kugelrohr distillation to afford a white solid (5.8 g, 88%). M.p. 49–52°C. ^{1}H-NMR (360 MHz, $CDCl_3$) δ 8.89–8.85 (m, 2H, pyr), 8.04–8.00 (m, 1H, pyr), 7.65–7.60 (m, 2H, pyr), 6.06–5.98 (m, 6H), 5.87–5.76 (m, 3H). ^{13}C-NMR (90 MHz, $CDCl_3$) δ 143.9, 140.6, 138.0, 131.3, 125.3.

2.4. Bis(dimethylamino)vinyl borane (7)

A solution of freshly distilled bis(dimethylamino)chloroborane (44.1 g, 0.328 mol) in distilled Et_2O/pentane (200 ml, 1:1 ratio) was cooled to −78°C with stirring. Vinylmagnesium chloride (1.5 M in THF, 219 ml, 0.328 mol) was added drop-wise over 2 h and the mixture was slowly warmed to 0°C and stirred for a further 12 h. The solid was removed through moisture protected filtering. The volatiles were distilled at atmospheric pressure and the residue was fractionally distilled under reduced pressure (52–53°C, 29–30 mmHg) to afford the product as a clear oil (38.0 g, 92%). Since the compound is extremely moisture sensitive, special care should be taken during distillation. ^{1}H-NMR (360 MHz, $CDCl_3$) δ 6.01 (dd, $J = 20.0$, 14.0 Hz, 1H), 5.73 (dd, $J = 14.0$, 4.3 Hz, 1H), 5.48 (dd, $J = 20.0$, 4.3 Hz, 1H), 2.70 (s, 12 H). ^{13}C-NMR (90 MHz, $CDCl_3$) δ 138.2 (br, C–B), 128.5, 40.5.

2.5. General procedure for the preparation of the TADDOL substituted vinylboronic esters

A solution of TADDOL (0.99 mmol) and ethyleneboronic anhydride pyridine complex (80 mg, 0.33 mmol) in toluene (10 ml) was placed in a 50 ml round bottom flask and fitted with Dean–Stark apparatus. The reaction was refluxed for 2 h, while the water/toluene azeotrope was trapped. After the reaction was cooled to r.t., the residue was further concentrated in vacuo (0.1 mmHg) to afford the vinyl–TADDOL–boronate (**9**).

2.6. Preparation of 11 and 12

A solution of the corresponding dibutyl vinyl boronate (1.55 mmol) and TADDOL (1.55 mmol) in xylene was refluxed for 2 h while the *n*-butanol/xylene azeotrope was trapped. The residue was concentrated in vacuo (0.1 mmHg) to afford the product. The product was used in the next reaction without further purification.

2.7. Di-n-butyl-(2-propenyl)-boronate

This compound has been prepared previously by Matteson [13] by a slightly different method. To a solution of freshly distilled trimethyl borate (8.73 g, 0.084 mol) in distilled THF (100 ml) was added drop-wise 2-propenylmagnesium bromide (2.2 M, 0.084 mol) at −78°C. The mixture was allowed to warm to r.t. over 2 h and then cooled to −78°C. Water (10 ml) and phenothiazine (0.03 g) were added and the addition of 3 M HCI (50 ml) followed. The mixture was allowed to warm to 0°C and stirred for 3 h. After all solid materials were dissolved, the two phases were separated. The aqueous phase was extracted with *n*-butanol (25 ml × 3) and the combined organic phases was washed with brine (20 ml × 3), then saturated sodium bicarbonate. The *n*-butanol/water azeotrope was first distilled off under reduced pressure and the residue was fractionally distilled under reduced pressure (3–4 mmHg, 83–86°C) to afford the product as a clear oil (12.4 g, 74%). ^{1}H-NMR (360 MHz, $CDCl_3$) δ 5.56 (s, 1H), 5.26 (s, 1H), 3.90–3.88 (m, 4H), 1.82 (s, 3H), 1.5–1.53 (m, 4H), 1.37-1.34 (m, 4H), 0.93–0.90 (m, 6H). ^{13}C-NMR (90 MHz, $CDCl_3$) δ 128.65, 124.16, 63.84, 33.84, 22.62, 18.98, 13.83.

2.8. Di-n-butyl-(a-styrenyl)-boronate

This compound has been prepared previously by Matteson [13] and coworkers by a slightly different method. This compound was prepared by a similar procedure to that described above. The crude product was purified by Kugelrohr distillation (85–90°C, 0.1

mmHg) to afford the product as a clear oil (80%). ^{1}H-NMR (360 MHz, $CDCl_3$) δ 7.41–7.20 (m, 5H), 5.93 (d, $J = 1.7$ Hz, 1H), 5.49 (d, $J = 1.7$ Hz, 1H), 3.85–3.82 (m, 4H), 1.55–1.54 (m, 4H), 1.35–1.32 (m, 4H), 0.91–0.89 (m, 6H). ^{13}C-NMR (90 MHz, $CDCl_3$) δ 141.50, 128.48, 127.49, 127.15, 126.34, 122.41, 64.27, 33.70, 18.95, 13.80.

2.9. Preparation of cycloadducts from **11**

A solution of **11** (974 mg, 1.55 mmol) and phenylhydroximic acid chloride (313 mg, 2.01 mmol) in freshly distilled THF (15 ml) was cooled to 0°C. Triethylamine (203 mg, 2.01 mmol) in THF (5 ml) was added dropwise over 30 min and stirred for 2 h. A white solid (triethylamine hydrochloride) was filtered through a glass frit under pressure (argon) and the filtrate was used in the next reaction.

2.10. Cycloadduct–diethanolamine complex

The solution above was treated with diethanolamine (210 mg, 2.0 mmol) at r.t. White solids began to form within 30 min. The reaction was stirred for 3 h and the solid was collected by vacuum filtration (ca. 85%). The product was pure enough for analytical purposes. The optically pure cycloadduct was obtained from recrystallization in acetone or toluene. M.p. 206–210°C. ^{1}H-NMR (360 MHz, $CDCl_3$) δ 7.61–7.55 (m, 2H), 7.45–7.31 (m, 3H), 6.11 (s, 1H), 4.15–3.89 (m, 4H), 3.58–3.38 (m, 3H), 3.02–2.81 (m, 3H), 1.22 (s, 3H). ^{13}C-NMR (90 MHz, $CDCl_3$) δ 157.12, 130.50, 129.51, 128.52, 126.53, 63.38, 63.19, 52.27, 52.16, 44.47, 23.82

2.11. Phenyl-5,5-methyl-pinanylboronat-Δ^2-isoxazoline

To a suspension of the diethanolamine complex above (363 mg, 1.40 mmol) and (+)-pinanediol (238 mg, 1.40 mmol) in Et_2O (6 ml) was added 4–5 drops of 6 M HCl at r.t. As the reaction proceeded, it became clear. The mixture was stirred for 3 h and the organic layer was vacuum-filtered through a short column packed with $MgSO_4$. The filtrate was concentrated under reduced pressure and further concentrated by vacuum pump (0.05 mmHg) to afford the product as a clear oil (455 mg, 100%). $[\alpha]_D^{20} = +10.63°$ ($c = 1.3$, CH_2Cl_2). B.p. 119–121°C (0.05 mmHg). ^{1}H-NMR (360 MHz, $CDCl_3$) δ 7.75–7.65 (m, 2H), 7.45–7.35 (m, 3H), 4.41 (dd, $J = 8.8$, 1.7 Hz, 1H), 3.52 (d, $J = 16.2$ Hz, 1H), 3.06 (d, $J = 16.2$ Hz, 1H), 2.41–2.30 (m, 1H), 2.29–2.25 (m, 1H), 2.13–2.10 (m, 1H), 1.95–1.93 (m, 2H), 1.46 (s, 3H), 1.40 (s, 3H), 1.30 (s, 3H), 1.15 (d, $J = 10.9$ Hz, 1H), 0.85 (s, 3H). ^{13}C-NMR (90 MHz, $CDCl_3$) δ 155.22, 130.11, 129.67, 128.55, 126.18, 86.98, 78.74, 51.18, 44.69, 39.34, 38.18, 35.21, 35.19, 30.19, 28.44, 27.00, 26.39, 23.93, 23.73, 23.68.

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