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Enantioselective 1,3-dipolar cycloadditions of nitrones with unsaturated aldehydes promoted by a recyclable tetraarylphosphonium supported imidazolidinone catalyst

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ABSTRACT

The tetraarylphosphonium supported chiral imidazolidinone catalyzes the enantioselective 1,3-dipolar cycloadditions of nitrones and α,β-unsaturated aldehydes to provide isoxazolidine aldehydes in good yields with excellent diastereo- and enantioselectivities. Most importantly, the tetraarylphosphonium supported imidazolidinone catalyst can be readily recovered and recycled for further transformations at least four cycles without observing significant decrease in yield and stereoselectivity.

1. Introduction

In the past few years, there has been a tremendous increase in the use of organocatalysts in asymmetric synthesis [1–5]. Among them, MacMillan's imidazolidinone catalyst has emerged as one of the most efficient organocatalysts for a variety of highly enantioselective reactions involving α,β-unsaturated aldehydes [6–10]. However, the recovery and recycling of the catalyst are still the issues that need to be addressed in this area. To solve this problem, recently several modified methods which allowed the recycling of the imidazolidinone catalyst by immobilizing MacMillan's catalyst onto ionic liquid, polymer, fluorous tag, or silica gel have been developed [11–24].

In a preliminary communication, we have reported the immobilization of chiral imidazolidinones on tetraarylphosphonium salts to afford the supported catalysts **1a–c** (Fig. 1) for the enantioselective Diels–Alder reaction of α,β-unsaturated aldehydes with dienes [25]. In continuation of our efforts in exploring the synthetic utility of the tetraarylphosphonium supported imidazolidinone in organic synthesis as well as developing practical organic transformations, herein we describe the extended application of the tetraarylphosphonium supported imidazolidinone catalyst **1a–c** to the 1,3-dipolar cycloadditions of α,β-unsaturated aldehydes with nitrones.

2. Experimental

2.1. General remarks

Reactions were monitored by TLC using precoated plates of silica gel HF254 (0.5 mm, Yantai, China). Column chromatography was performed with a silicagel column (200–300 mesh, Yantai, China). NMR spectra were recorded on Varian Unity Inova 600 spectrometer (^{1}H at 600 MHz and ^{13}C at 150 MHz) or WIPM 400 spectrometer (^{1}H at 400 MHz and ^{13}C at 100 MHz). IR spectra were recorded on an IR-spectrum one (PE) spectrometer. HPLC were performed with Dionex UltiMate 3000 and equipped with a chiral column (Chiralcel OJ-RH, Daicel) using CH_3OH/H_2O as an eluent. A UV detector (UVD-3000) was used for the peak detection.

2.2. General procedure for the 1,3-dipolar cycloadditions

To a solution of the tetraarylphosphonium supported imidazolidinone catalyst **1a** (438 mg, 0.6 mmol) in CH_3NO_2 (30 mL) and H_2O (27 μL) was added HBF_4 (40 μL, 0.6 mmol) and *N*-benzyl-*C*-phenyl nitrone (633 mg, 3 mmol). After cooling the solution to 0 °C,

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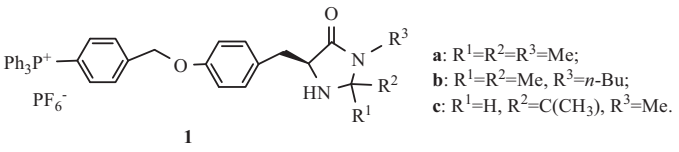


1

Fig. 1. Structure of the tetraarylphosphonium supported chiral imidazolidinones.

E-crotonaldehyde (2.5 mL, 30 mmol) was added, and then the mixture was stirred for 36 h at this temperature. The resulting solution was then evaporated under vacuum. The residue was dissolved in the minimum amount of CH_2Cl_2 (2 mL) and poured in Et_2O (25 mL), and then filtered. The filtrate was concentrated under vacuum and purified by silica gel column chromatography (*n*-hexane/EtOAc, 50:1, v/v) to afford the desired products, in which the *endo*/*exo* ratio was determined by ^{1}H NMR. The precipitate in the sand-core funnel was dried under vacuum and reused in further reactions as the recovered catalyst. The recovered catalyst was examined by ^{1}H NMR spectroscopy, which showed unchanged after each recovery.

Spectroscopic data of the 1,3-dipolar cycloaddition products:

(4*S*, 5*R*)-2-Benzyl-5-methyl-3-phenylisoxazolidine-4-carbaldehyde (2a): IR (NaCl): υ 2928, 1720, 1603, 1495, 1455, 1373 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 9.71 (*d*, *J* = 2.3 Hz, 1H, C**H**O), 7.37–7.15(m, 10H, Ar**H**), 4.49–4.46 (*m*, 1H, C**H**CH$_3$), 4.10 (*d*, *J* = 7.8 Hz, 1H, C_6H_5C**H**), 3.95 (*d*, *J* = 14.3 Hz, 1H, C_6H_5C**H**$_2$), 3.77 (*d*, *J* = 14.3 Hz, 1H, C_6H_5C**H**$_2$), 3.06–3.03 (*m*, 1H, C**H**CHO), 1.43 (*d*, *J* = 6.2 Hz, 3H, **CH$_3$**); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 198.6, 138.2, 137.2, 129.0, 128.9, 128.5, 128.3, 127.7, 127.2, 73.6, 71.5, 71.2, 59.5, 21.1. Enantiomeric ratio was determined by HPLC using Chiracel OJ-RH column after reduction with $NaBH_4$/MeOH (80:20 MeOH/H_2O, 1 mL/min flow rate), *endo* isomers t_R = 7.1 min (major enantiomer) and 6.4 min (minor enantiomer).

(4*S*, 5*R*)-2-Benzyl-4-formyl-5-methyl-3-(4-chlorophenyl) isoxazolidine (2b): IR (NaCl): υ 2975, 1723, 1493, 1455, 1372, 1090 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 9.65 (*d*, *J* = 2.1 Hz, 1H, C**H**O), 7.26–7.16 (*m*, 9H, Ar**H**), 4.45–4.39 (*m*, 1H, C**H**ON), 4.07 (*d*, *J* = 7.4 Hz, 1H, ClC_6H_4C**H**), 3.87 (*d*, *J* = 14.2 Hz, 1H, C_6H_5C**H**$_2$), 3.75 (*d*, *J* = 14.2 Hz, 1H, C_6H_5C**H**$_2$), 2.92–2.95 (*m*, 1H, C**H**CHO), 1.38 (*d*, *J* = 6.2 Hz, 3H, C**H$_3$**); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 198.3, 137.3, 137.0, 133.8, 129.1, 128.9, 128.3, 127.3, 122.0, 73.6, 71.6, 70.1, 59.7, 21.0. Enantiomeric ratio was determined by HPLC using Chiracel OJ-RH column after reduction with $NaBH_4$/MeOH (80:20 MeOH/H_2O, 1 mL/min flow rate), *endo* isomers t_R = 11.2 min (major enantiomer) and 13.2 min (minor enantiomer).

(4*S*, 5*R*)-2-Benzyl-4-formyl-5-methyl-3-(4-methoxyphenyl) isoxazolidine (2c): IR (NaCl): υ 2836, 1723, 1513, 1455, 1303, 1032 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 9.64 (*d*, *J* = 2.3 Hz, 1H, C**H**O), 7.26–7.10 (*m*, 7H, Ar**H**), 6.80 (*d*, *J* = 8.8 Hz, 2H, orthoC_6**H$_4$**OCH$_3$), 4.44–4.38 (*m*, 1H, C**H**ON), 3.98 (*d*, *J* = 8.0 Hz, 1H, $CH_3OC_6H_5$C**H**), 3.90 (*d*, *J* = 14.5 Hz, 1H, C_6H_5C**H**$_2$), 3.68–3.65 (*m*, 4H, OC**H$_3$**, C_6H_5C**H**$_2$), 2.95–2.99 (*m*, 1H, C**H**CHO), 1.39 (*d*, *J* = 6.2 Hz, 3H, C**H$_3$**); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 198.8, 159.6, 137.5, 129.9, 128.9, 128.8, 128.5, 128.2, 127.1, 114.4, 73.4, 71.5, 70.9, 59.3, 21.2. Enantiomeric ratio was determined by HPLC using Chiracel OJ-RH column after reduction with $NaBH_4$/MeOH (80:20 MeOH/H_2O, 1 mL/min flow rate), *endo* isomers t_R = 7.3 min (major enantiomer) and 9.5 min (minor enantiomer).

(4*S*, 5*R*)-2-Benzyl-4-formyl-5-methyl-3-(4-methylphenyl) isoxazolidine (2d): IR (NaCl): υ 2925, 1724, 1514, 1454, 1372, 1076 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$, ppm): δ 9.69 (*d*, *J* = 2.4 Hz, 1H, C**H**O), 7.26–7.08 (*m*, 9H, Ar**H**), 4.47–4.43 (*m*, 1H, C**H**ON), 4.02 (*d*, *J* = 7.9 Hz, 1H, $CH_3C_6H_5$C**H**), 3.92 (*d*, *J* = 14.4 Hz, 1H, C_6H_5C**H**$_2$), 3.72 (*d*, *J* = 14.4 Hz, 1H, C_6H_5C**H**$_2$), 3.03–3.00 (*m*, 1H, C**H**CHO), 2.27 (*s*, 3H, C_6H_4C**H**$_3$), 1.42 (*d*, *J* = 6.2 Hz, 3H, OCHC**H$_3$**); ^{13}C NMR (150 MHz, $CDCl_3$, ppm): δ 198.5, 137.9, 137.3, 135.1, 129.5, 128.4, 128.1, 127.5, 127.0, 73.4, 71.5, 71.1, 59.3, 21.0, 20.9. Enantiomeric ratio was determined by HPLC using Chiracel OJ-RH column after reduction with $NaBH_4$/MeOH (80:20 MeOH/H_2O, 1 mL/min flow rate), *endo* isomers t_R = 8.5 min (major enantiomer) and 10.0 min (minor enantiomer).

(4*S*, 5*R*)-2-Benzyl-4-formyl-5-methyl-3-(2-napthyl) isoxazolidine (2e): IR (NaCl): υ 2866, 1723, 1601, 1496, 1454, 1125 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$, ppm): δ 9.72 (*d*, *J* = 1.9 Hz, 1H, C**H**O), 7.77–7.20 (*m*, 12H, Ar**H**), 4.52–4.48 (*m*, 1H, C**H**ON), 4.24 (*d*, *J* = 7.9 Hz, 1H, C**H**NO), 3.96 (*d*, *J* = 14.3 Hz, 1H, C_6H_5C**H**$_2$), 3.80 (*d*, *J* = 14.3 Hz, 1H, C_6H_5C**H**$_2$), 3.13–3.10 (*m*, 1H, C**H**CHO), 1.45 (*d*, *J* = 6.2 Hz, 3H, OCHC**H$_3$**); ^{13}C NMR (150 MHz, $CDCl_3$, ppm): δ 198.4, 137.1, 135.8, 133.4, 133.2, 128.9, 128.6, 128.2, 127.9, 127.7, 127.2, 126.9, 126.4, 126.2, 124.9, 73.7, 71.5, 71.3, 59.7, 20.9. Enantiomeric ratio was determined by HPLC using Chiracel OJ-RH column after reduction with $NaBH_4$/MeOH (70:30 MeOH/H_2O, 1 mL/min flow rate), *endo* isomers t_R = 69.7 min (major enantiomer) and 76.0 min (minor enantiomer).

(4*S*, 5*R*)-2-Benzyl-4-formyl-5-propyl-3-phenylisoxazolidine (2f): IR (NaCl): υ 2872, 1725, 1495, 1455, 1377, 1047 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 9.70 (*d*, *J* = 2.6 Hz, 1H, C**H**O), 7.35–7.13 (*m*, 10H, Ar**H**), 4.29–4.21 (*m*, 1H, C**H**CH$_2$CH$_2$CH$_3$), 4.06 (*d*, *J* = 7.8 Hz, 1H, C_6H_5C**H**), 3.93 (*d*, *J* = 14.3 Hz, 1H, C_6H_5C**H**$_2$), 3.74 (*d*, *J* = 14.3 Hz, 1H, C_6H_5C**H**$_2$), 3.08–3.04 (*m*, 1H, C**H**CHO), 1.94–1.85 (*m*, 1H, CHC**H**$_2$CH$_2$CH$_3$), 1.60–1.54 (*m*, 1H, CHC**H**$_2$CH$_2$CH$_3$), 1.40–1.24 (*m*, 2H, CH$_2$C**H**$_2$CH$_3$), 0.87–0.84 (*t*, *J* = 7.3 Hz, 3H, C**H$_3$**); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 198.7, 138.2, 137.3, 128.9, 128.4, 128.2, 128.1, 127.6, 127.1, 77.3, 71.0, 70.4, 59.3, 37.6, 19.2, 13.9. Enantiomeric ratio was determined by HPLC using Chiracel OJ-RH column after reduction with $NaBH_4$/MeOH (70:30 MeOH/H_2O, 1 mL/min flow rate), *endo* isomers t_R = 7.3 min (major enantiomer) and 6.0 min (minor enantiomer).

(*S*)-2-Benzyl-4-formyl-3-phenylisoxazolidine (2g): IR (NaCl): υ 2875, 1722, 1495, 1455, 1047 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ 9.60 (*d*, *J* = 3.7 Hz, 1H, C**H**O), 7.35–7.11 (*m*, 10H, Ar**H**), 4.12–4.05 (*m*, 2H, C**H$_2$**ON), 3.93 (*d*, *J* = 7.4 Hz, 1H, C_6H_5C**H**), 3.86 (*d*, *J* = 14.1 Hz, 1H, C_6H_5C**H**$_2$), 3.65 (*d*, *J* = 14.1 Hz, 1H, C_6H_5C**H**$_2$), 3.26–3.24 (*m*, 1H, C**H**CHO); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ 198.9, 138.3, 137.4, 129.1, 128.8, 128.4, 128.3, 127.9, 127.4 70.4, 65.9, 64.4, 59.8. Enantiomeric ratio was determined by HPLC using Chiracel OJ-RH column after reduction with $NaBH_4$/MeOH (70:30 MeOH/H_2O, 1 mL/min flow rate), *endo* isomers t_R = 18.6 min (major enantiomer) and 20.8 min (minor enantiomer).

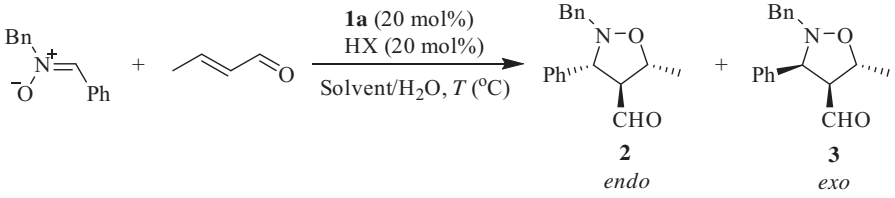
3. Results and discussion

3.1. Catalytic activity of catalyst **1a–c** in the 1,3-dipolar cycloadditions

To evaluate the capacity of the tetraarylphosphonium supported chiral imidazolidinones **1a–c** in the 1,3-dipolar cycloadditions, the synthesis of the isoxazolidine aldehyde via *N*-benzyl-*C*-phenyl nitrone with *E*-crotonaldehyde was chosen as a model reaction. Then we sought to establish optimal reaction conditions that would furnish the isoxazolidine aldehyde products **2** and **3** in good yield and excellent enantioselectivity.

As illustrated in Table 1, the choice of solvent is found to have a significant impact on the outcome of the reaction, not only in terms of yield, but also involve the enantioselectivity (Table 1, entries 1–8). The reactions were carried out in the presence of 20 mol% **1a**/HBF_4 combinations in different solvent system at 4–25 °C for 24 h. Among the solvents examined, CH_3NO_2/H_2O provided the best result (entry 8, 82% yield, 94/6 *endo*/*exo*, *endo* 88% ee). Therefore, CH_3NO_2/H_2O was chosen as the solvent system to further

Table 1
Optimization of 1,3-dipolar cycloaddition reaction conditions between *E*-crotonaldehyde and nitrone.



Entry	Catalyst	Solvent	HX	*T* (°C)	Time (h)	Yield[b] (%)	*endo*/*exo*[c]	*endo* ee[d] (%)
1	**1a**	$CHCl_3$	HBF_4	4–25[a]	24	73	90/10	70
2	**1a**	$CHCl_2$	HBF_4	4–25	24	68	75/25	63
3	**1a**	THF	HBF_4	4–25	24	44	78/22	68
4	**1a**	CH_3CN	HBF_4	4–25	24	65	91/9	76
5	**1a**	DMF	HBF_4	4–25	24	27	82/18	82
6	**1a**	H_2O	HBF_4	4–25	24	40	74/26	50
7	**1a**	Toluene	HBF_4	4–25	24	65	80/20	39
8	**1a**	CH_3NO_2	HBF_4	4–25	24	82	94/6	88
9	**1a**	CH_3NO_2	CF_3SO_3H	4–25	24	53	63/37	43
10	**1a**	CH_3NO_2	HCl	4–25	24	40	81/19	77
11	**1a**	CH_3NO_2	CF_3COOH	4–25	24	51	88/12	72
12	**1a**	CH_3NO_2	$HClO_4$	4–25	24	71	91/9	86
13	**1a**	CH_3NO_2	HPF_6	4–25	24	77	93/7	80
14	**1a**	CH_3NO_2	CH_3COOH	4–25	24	32	84/16	72
15	**1a**	CH_3NO_2	HBF_4	0	12	Trace	—	—
16	**1a**	CH_3NO_2	HBF_4	0	24	75	91/9	89
17	**1a**	CH_3NO_2	HBF_4	0	36	89	94/6	94
18	**1a**	CH_3NO_2	HBF_4	0	48	89	93/7	93
19	**1a**	CH_3NO_2	HBF_4	−20	72	65	90/10	92
20	**1b**	CH_3NO_2	HBF_4	0	36	86	92/8	76
21	**1c**	CH_3NO_2	HBF_4	0	36	90	70/30	45
22	**1a**	CH_3NO_2	HBF_4[e]	0	36	72	88/12	94
23	**1a**	CH_3NO_2	HBF_4[f]	0	36	77	92/8	92
24	**1a**	CH_3NO_2	HBF_4[g]	0	36	86	94/6	94
25	**1a**	CH_3NO_2	HBF_4[h]	0	36	85	90/10	92
26	**1a**	CH_3NO_2	HBF_4[i]	0	36	82	90/10	92

[a] Substrates were added at 4 °C and then the mixture was stirred for 24 h while the temperature was allowed to rise slowly to 25 °C.
[b] Isolated yield of a mixture of *endo* and *exo* isomers.
[c] *Exo*/*endo* ratios were determined by ^1H NMR analysis.
[d] ee values were determined by HPLC of alcohol after reduction of the formyl group.
[e] 10 mol% catalyst.
[f] 30 mol% catalyst.
[g] 2nd run.
[h] 3rd run.
[i] 4th run.

optimize the reaction conditions by screening different acids as reaction co-catalysts.

Seven types of protic acids were screened as the reaction co-catalysts (Table 1, entries 8–14). Among the protic acids examined, it was found that better *endo*/*exo* selectivities (94/6, 91/9, 93/7) were obtained when HBF_4, $HClO_4$ and HPF_6 used as reaction co-catalysts (entries 8, 12 and 13). In contrast, relatively high yield and excellent *endo* ee were produced when HBF_4 was selected (entry 8, 82% and 88%). Thus, HBF_4 was chosen as the optimal reaction co-catalyst in further reactions.

Next, the effects of reaction temperature and time, universally crucial factors, in the 1,3-dipolar cycloaddition was investigated (Table 1, entries 8, 15–19). It is manifest that 0 °C and 36 h is the best temperature and time for the reaction, which resulted in 89% yield, 94/6 *endo*/*exo* and 94% *endo* ee.

Furthermore, the influence of the catalyst loading in the 1,3-dipolar cycloaddition was also examined under the condition of 0 °C for 36 h (Table 1, entries 17, 22 and 23). The best amount of catalyst used was 20 mol%, as higher or lower catalyst loadings seemed to be detrimental either for yield or stereoselectivity. So the optimal reaction condition of the 1,3-dipolar cycloaddition with **1a** as catalyst is CH_3NO_2/H_2O as solvent system, HBF_4 as co-catalyst, 0 °C for 36 h and 20 mol% catalyst amount.

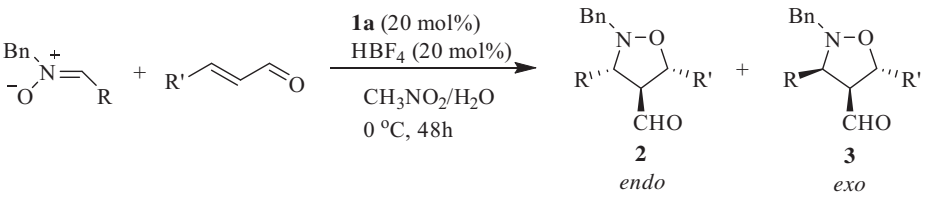
With the optimized reaction condition in hand, the tetraarylphosphonium supported chiral imidazolidin-4-ones **1b** and **1c** were also evaluated for the 1,3-dipolar cycloaddition to compare with the use of **1a** (entries 20 and 21). The chemical yields by the use of the catalysts **1b** and **1c** were comparable to **1a**, but the diastereoselectivity and enantioselectivity were lower. So the catalyst **1a** is the best one among them.

Overall, the target products **2** and **3** were obtained in 89% yield, 94/6 *endo*/*exo* and 94% *endo* ee by **1a**, which is comparable to the result obtained from the use of non-supported imidazolidinone [10]. For instance, the same excellent *endo* enantioselectivity of 94% was obtained by employing either Macmillan's imidazolidinone catalyst or the tetraaryphosphonium supported catalyst.

3.2. Recycling and reuse of catalyst *1a*

Isolation of the products by simple precipitation and filtration was quite easy when using the tetraaryphosphonium supported chiral imidazolidinone catalyst. Catalyst recycling experiments could also be readily performed. As shown in Table 1 (entries 17, 24–26), no significant decrease in catalytic activity is observed when the tetraaryphosphonium supported catalyst **1a** was recovered and reused in further reactions. Excellent diastereoselectivity

Table 2
Scope of catalyst **1a** in the 1,3-dipolar cycloadditions.



Entry	*R*	*R'*	Products	Yield[a] (%)	*endo*/*exo*[b]	*endo* ee[c] (%)
1	Ph	Me	**2a** + **3a**	89	94/6	94
2	4-ClPh	Me	**2b** + **3b**	72	90/10	90
3	4-OMePh	Me	**2c** + **3c**	78	93/7	88
4	4-MePh	Me	**2d** + **3d**	79	91/9	85
5	2-Naphthyl	Me	**2e** + **3e**	81	90/10	92
6	Ph	*n*-Pr	**2f** + **3f**	72	94/6	97
7	Ph	H	**2g** + **3g**	77	94/6	88

[a] Isolated yield of a mixture of *endo* and *exo* isomers.
[b] *Exo*/*endo* ratios were determined by ^{1}H NMR analysis.
[c] ee values were determined by HPLC of alcohol after reduction of the formyl group.

(90/10) and enantioselectivity (92% *endo* ee) of the corresponding products were obtained even in the 4th cycle, making catalyst **1a** synthetically efficient and useful (entry 26).

3.3. Scope of catalyst *1a*

The scope of the 1,3-dipolar cycloadditions between α,β-unsaturated aldehydes and various nitrones catalyzed by **1a** was investigated (Table 2). The reaction appears quite general with respect to the nitrone structure (entries 1–5, 72–88% yield, 90/10 to 94/6 *endo*/*exo*, 85–97% *endo* ee). Changes in the structure of the dipolarophile were also well tolerated; *E*-crotonaldehyde, *E*-2-hexenal and acrolein provided isoxazolidine aldehydes in good yields with excellent diastereo- and enantioselectivities (entries 1, 6 and 7).

4. Conclusions

In conclusion, we have developed an efficient method for the enantioselective 1,3-dipolar cycloadditions by using the tetraarylphosphonium supported imidazolidinone catalyst, providing the desired products in good yields with excellent diastereo- and enantioselectivities. It is worth mentioning that the tetraarylphosphonium supported imidazolidinone catalyst can be readily recovered and recycled for further transformations at least four cycles without observing significant decrease in yield and stereoselectivity.

Acknowledgments

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