

# Efficient synthesis of symmetrical phthalate and maleate diesters using phosphinite ionic liquids

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**Abstract** Symmetrical dialkyl phthalates and maleates were synthesized using phosphinite ionic liquid as a catalyst and reaction medium. The results indicated that phosphinite ionic liquid shows better catalytic and reusable performance without using any acid or base catalyst. Under the optimum conditions, using 1-methyl-3-(4-phosphinitebutyl) imidazolium chloride as catalyst, the conversion of phthalic and maleic anhydrides to the corresponding diesters of primary and secondary alcohols was occurred in 72–85% yields. The diesters of tertiary alcohols and phenols could not be prepared by this method. A kind of widely used plasticizer, dioctyl phthalate, was prepared in good yield under these conditions. The ionic liquid could be reused three times after easy separation from the products without any disposal.

**Keywords** Symmetrical maleate diester · Symmetrical phthalate diester · Phosphinite ionic liquid · Maleic anhydride · Phthalic anhydride

## Introduction

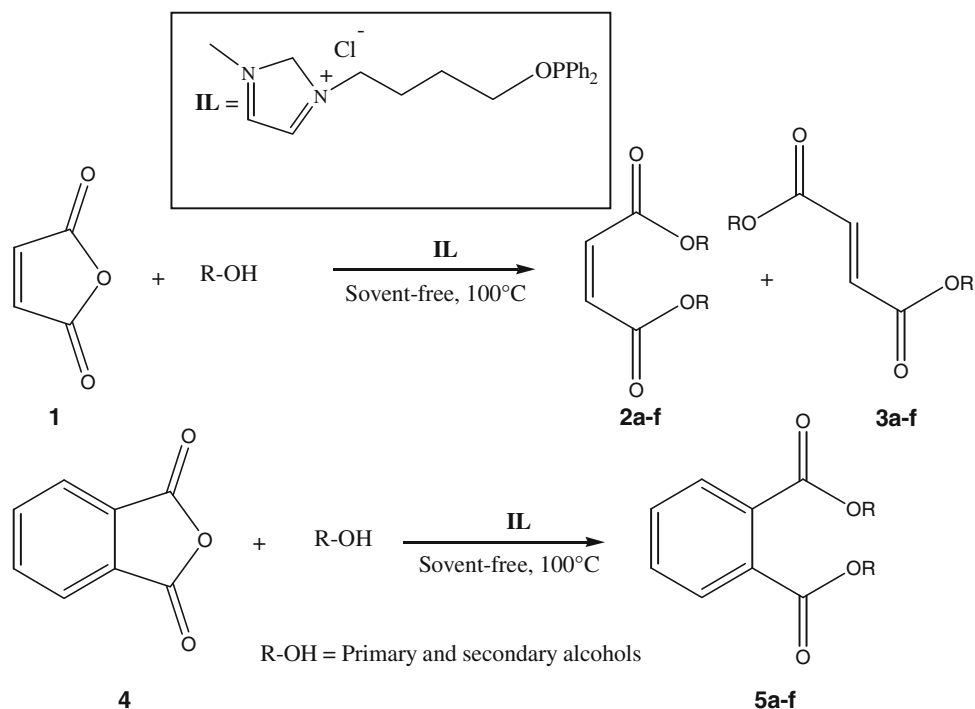
Esters are important organic compounds which are used as an intermediate in the synthesis of fine chemicals, drugs, plasticizers, chiral-auxiliaries, agrochemicals [1]. Esterification is an industrially important reaction which conventionally catalyzed by an environmentally hazardous chemical sulphuric acid [2]. Many other catalysts such as

super acid [3], zeolites [4], heteropolyacids (HPA) [5] and solid acids M (IV) tangestates and phosphates [6] have been used for the synthesis of esters. Sejidov and co-workers [7] used solid heterogeneous acid catalysts for the esterification reaction under solvent-free conditions. The applications of these catalysts cause some problems such as corrosion, loss of catalyst, and environment problems. Therefore, development of more efficient catalysts will be interesting and useful. Li et al. [8] reported the synthesis of dioctyl phthalate using acid functionalized ionic liquid as catalyst.

The room temperature ionic liquids (RTIL's) are environmental friendly solvents and catalysts which have adjustable physical and chemical properties. These compounds obtained broad attention of researchers from various fields such as synthesis, catalysis, separation and electrochemistry [9–12]. There are many articles on the subject of functionalized ionic liquid (FIL) used in different organic reactions [13–15]. Recently, we reported the dizotization reaction and subsequent synthesis of azo dyes using nitrite ester functionalized ionic liquid, IL-ONO [16]. Iranpoor and co-workers used phosphinite ionic liquid as a reusable reaction medium and Pd (II) ligand in Heck reactions of aryl halides with styrene and *n*-butyl acrylate [17]. In our previous works, we have used phosphinite ionic liquid as Lewis base catalyst and reaction medium for the synthesis of coumarins and 3,4-dihydropyrimidin-2(1*H*)-(thio)ones [18, 19]. We examined the ability of this ionic liquid as a possible Lewis base catalyst in the reaction of maleic anhydride and alcohols for the possible preparation of related acrylate esters [20]. Under different conditions, acrylate ester was not synthesized using this IL and the related maleate diester was prepared. In this work, we wish to report the esterification of maleic and phthalic anhydrides with various primary and secondary alcohols in

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**Scheme 1** Synthesis of symmetrical diesters of dialkyl maleates and phthalates using IL-OPPh<sub>2</sub>



IL-OPPh<sub>2</sub> as catalyst and reaction medium without using any solvent and catalyst (Scheme 1). The symmetrical maleate and phthalate diesters were prepared in good yields under these conditions.

## Experimental

### General

All reagents were purchased from Merck Company and used without further purification. Infrared spectra were recorded in KBr and were determined on a Perkin-Elmer FT-IR spectrometer. <sup>1</sup>H NMR spectra were recorded on a Bruker Avance AC-400 MHz using DMSO-d<sub>6</sub>, acetone-d<sub>6</sub> or CDCl<sub>3</sub> as the deuterated solvents and TMS as internal standard. Elemental analyses were carried out on a Perkin-Elmer 240C elemental analyzer and are reported in percent atomic abundance.

### Synthesis of dialkyl maleate and phthalate diesters using IL-OPPh<sub>2</sub>: general procedure

Anhydride (0.2 mol), alcohol (0.42 mol) and excess amount of the freshly prepared phosphinite ionic liquid were stirred at 100 °C. The completion of reaction was monitored by TLC using (EtOAc/petroleum 2:5) as eluent. After completion of the reaction as indicated by TLC, the product and unreacted materials were washed by diethyl ether (3 × 8 ml) and isolated from ionic liquid. The diethyl

ether was removed under reduced pressure at room temperature and the residue was purified by column chromatography to afford pure desired products.

### Selected spectroscopic data

Cis di-*n*-butyl maleate (**2a**): FT-IR (KBr, cm<sup>-1</sup>): 1,728 (C=O), 1,646 (C=C), 1,231 (C-O). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm): 0.98 (t, 6H, -Me, *J* = 7.72 Hz), 1.32 (m, 4H, -CH<sub>2</sub>), 1.36 (m, 4H, -CH<sub>2</sub>), 4.20 (t, 4H, -OCH<sub>2</sub>, *J* = 7.85 Hz), 6.86 (s, 2H, -CH=CH-). Anal. Calcd for C<sub>12</sub>H<sub>20</sub>O<sub>4</sub>: C, 63.14; H, 8.83%. Found (%): C, 63.15; H, 8.84.

Trans di-*n*-butyl maleate (**3a**): FT-IR (KBr, cm<sup>-1</sup>): 1,723 (C=O), 1,638 (C=C), 1,224 (C-O). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm): 1.02 (t, 3H, -Me, *J* = 7.75 Hz), 1.30 (m, 2H, -CH<sub>2</sub>), 1.33 (m, 2H, -CH<sub>2</sub>), 4.15 (t, 2H, -OCH<sub>2</sub>, *J* = 7.46 Hz), 6.92 (s, 2H, -CH=CH-). Anal. Calcd for C<sub>12</sub>H<sub>20</sub>O<sub>4</sub>: C, 63.14; H, 8.83%. Found (%): C, 63.18; H, 8.85.

Di-*n*-butyl phthalate (**5a**): FT-IR (KBr, cm<sup>-1</sup>): 1,719 (C=O), 1,645 (C=C), 1,592 (C=C), 1,228 (C-O). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm): 1.05 (t, 6H, -Me, *J* = 7.85 Hz), 1.35 (m, 4H, -CH<sub>2</sub>), 1.39 (m, 4H, -CH<sub>2</sub>), 4.09 (t, 4H, -OCH<sub>2</sub>, *J* = 7.58 Hz), 7.54 (m, 2H, Ar-H), 7.73 (m, 2H, Ar-H). Anal. Calcd for C<sub>16</sub>H<sub>22</sub>O<sub>4</sub>: C, 69.04; H, 7.97%. Found (%): C, 69.23; H, 7.98.

Cis di-iso-butyl maleate (**2b**): FT-IR (KBr, cm<sup>-1</sup>): 1,719 (C=O), 1,639 (C=C), 1,228 (C-O). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ (ppm): 0.99 (t, 6H, 2Me, *J* = 7.22 Hz), 1.03

(d, 6H, 2Me,  $J = 7.35$  Hz), 1.10 (m, 4H, 2 CH<sub>2</sub>), 4.02 (m, 2H, –OCH), 6.27 (s, 2H, –CH=CH–). Anal. Calcd for C<sub>12</sub>H<sub>20</sub>O<sub>4</sub>: C, 63.14; H, 8.83%. Found (%): C, 63.16; H, 8.85.

Trans di-iso-butyl maleate (**3b**): FT-IR (KBr, cm<sup>-1</sup>): 1,724 (C=O), 1,635 (C=C), 1,217 (C–O). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 0.97 (t, 6H, 2Me,  $J = 7.25$  Hz), 1.01 (d, 6H, 2Me,  $J = 7.36$  Hz), 1.09 (m, 4H, 2 CH<sub>2</sub>), 4.02 (m, 2H, –OCH), 6.31 (s, 2H, –CH=CH–). Anal. Calcd for C<sub>12</sub>H<sub>20</sub>O<sub>4</sub>: C, 63.14; H, 8.83%. Found (%): C, 63.2; H, 8.87.

Di-iso-butyl phthalate (**5b**): FT-IR (KBr, cm<sup>-1</sup>): 1,718 (C=O), 1,631 (C=C), 1,215 (C–O). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 0.95 (t, 6H, 2Me,  $J = 7.33$  Hz), 0.99 (d, 6H, 2Me,  $J = 7.39$  Hz), 1.10 (m, 4H, 2 CH<sub>2</sub>), 4.09 (m, 2H, –OCH), 7.28–7.33 (m, 2H, Ar–H), 7.35–7.38 (m, 2H, Ar–H). Anal. Calcd for C<sub>16</sub>H<sub>22</sub>O<sub>4</sub>: C, 69.04; H, 7.97%. Found (%): C, 69.15; H, 8.01.

Cis di-cyclohexyl maleate (**2f**): FT-IR (KBr, cm<sup>-1</sup>): 1,721 (C=O), 1,648 (C=C), 1,230 (C–O). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 1.05 (m, 20H, cyclohexyl protons), 4.09 (m, 2H, –OCH<sub>2</sub>), 6.31 (s, 2H, –CH=CH–). Anal. Calcd for C<sub>16</sub>H<sub>24</sub>O<sub>4</sub>: C, 68.54; H, 8.63%. Found (%): C, 68.56; H, 8.64.

Trans di-cyclohexyl maleate (**3f**): FT-IR (KBr, cm<sup>-1</sup>): 1,726 (C=O), 1,650 (C=C), 1,233 (C–O). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 1.02 (m, 20H, cyclohexyl protons), 3.98 (m, 2H, –OCH<sub>2</sub>), 6.29 (s, 2H, –CH=CH–). Anal. Calcd for C<sub>16</sub>H<sub>24</sub>O<sub>4</sub>: C, 68.54; H, 8.63%. Found (%): C, 68.57; H, 8.66.

Di-cyclohexyl phthalate (**5f**): FT-IR (KBr, cm<sup>-1</sup>): 1,732 (C=O), 1,641 (C=C), 1,227 (C–O). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 1.04 (m, 20H, cyclohexyl protons), 4.11 (m, 2H, –OCH<sub>2</sub>), 7.21 (s, 2H, Ar–H), 7.32 (s, 2H, Ar–H). Anal. Calcd for C<sub>20</sub>H<sub>26</sub>O<sub>4</sub>: C, 72.70; H, 7.93%. Found (%): C, 72.73; H, 7.95.

Cis dibenzyl maleate (**2g**): FT-IR (KBr, cm<sup>-1</sup>): 1,732 (C=O), 1,638 (C=C), 1,225 (C–O). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 5.17 (s, 4H, –OCH<sub>2</sub>), 6.32 (s, 2H, –CH=CH–), 7.34–7.41 (m, 10H, Ar–H). Anal. Calcd for C<sub>18</sub>H<sub>16</sub>O<sub>4</sub>: C, 72.96; H, 5.44%. Found (%): C, 72.94; H, 5.45.

Trans dibenzyl maleate (**3g**): FT-IR (KBr, cm<sup>-1</sup>): 1,725 (C=O), 1,640 (C=C), 1,231 (C–O). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 5.09 (s, 4H, –OCH<sub>2</sub>), 6.30 (s, 2H, –CH=CH–), 7.31–7.38 (m, 10H, Ar–H). Anal. Calcd for C<sub>18</sub>H<sub>16</sub>O<sub>4</sub>: C, 72.96; H, 5.44%. Found (%): C, 72.97; H, 5.46.

Dibenzyl phthalate (**5g**): FT-IR (KBr, cm<sup>-1</sup>): 1,718 (C=O), 1,638 (C=C), 1,229 (C–O). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 5.21 (s, 4H, –OCH<sub>2</sub>), 7.21–7.25 (m, 2H, Ar–H), 7.27–7.31 (m, 2H, Ar–H), 7.36–7.43 (m, 10H, Ar–H). Anal. Calcd for C<sub>22</sub>H<sub>18</sub>O<sub>4</sub>: C, 76.29; H, 5.24%. Found (%): C, 76.33; H, 5.27.

## Results and discussions

According to the report of Li et al. [8], dioctyl phthalate can be prepared via the esterification of phthalic anhydride with octyl alcohol in the presence of acidic functionalized ionic liquid. They also used the neutral ionic liquids for this reaction and lower yield of dioctyl phthalate was prepared under these conditions. According to our earlier reports, phosphinite ionic liquid, IL-OPPh<sub>2</sub>, can act as a weak Lewis base catalyst [18, 19]. So, as described in “Introduction”, the catalytic behavior of this ionic liquid in the well-known acid catalyzed esterification reaction surprised us. To investigate the scope and limitation of IL-OPPh<sub>2</sub> for the esterification reaction, various alcohols were used in the reaction with maleic and phthalic anhydrides. Phosphinite ionic liquid was synthesized according to Iranpoor and co-worker’s reported work [17]. To examine its activity, the esterification reaction of maleic anhydride was reacted with two equivalent of benzyl alcohol at room temperature in the absence of any additives. The experimental results revealed that this ionic liquid, exhibits a very high activity. During the course of our further optimization of the reaction conditions, when using one half equivalent of ionic liquid in toluene as solvent, the reactions were generally completed in a matter of hours, but the time, as expected, was inversely proportional to the temperature. A toluene reflux temperature was found to be optimal. We also examined this procedure in the presence of phosphinite ionic liquid without any solvent. Good yield of diester product was isolated at 100 °C under these conditions. Thus, the optimized reaction conditions for this esterification reaction are the phosphinite ionic liquid (excess amount) and 100 °C for 12–15 h. To study the versatility of this procedure, the esterification of maleic and phthalic anhydrides with phenol, and various primary, secondary and tertiary alcohols was examined. The results are listed in Table 1. It shows that the yields of diesters from primary and secondary alcohols with both the phthalic and maleic anhydrides were satisfactory under the present reaction conditions. For the primary and secondary alcohols such as *n*-octanol, *n*-butanol, benzyl alcohol, 1-phenyl ethanol, isopropanol and cyclohexanol, excellent yields of diester products were isolated from the reaction with both of maleic and phthalic anhydrides under the optimized reaction conditions (Table 1). On the other hand, the esterification of the relatively hindered tertiary alcohols such as *tert*-butanol and 2-phenyl-2-propanol with both of maleic and phthalic anhydride was not preceded significantly and only the reactants were isolated after 24 h stirring of the reaction mixture under optimized conditions. We also examined the reaction of phenol and *p*-cresol with maleic and phthalic anhydrides and no ester or diester products were prepared after 24 h stirring of the reaction mixture

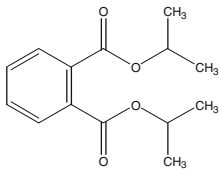
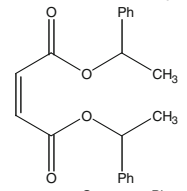
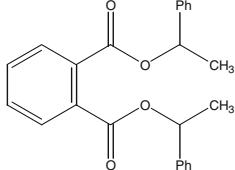
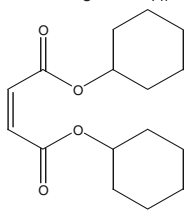
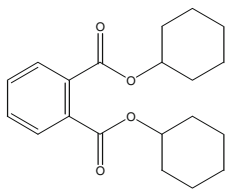
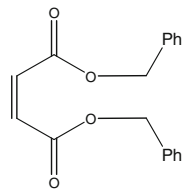
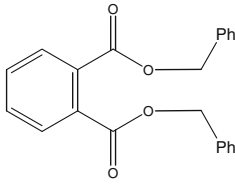
**Table 1** Synthesis of dialkyl maleate and phthalate diesters using phosphinite ionic liquid

Entry	Product <sup>a</sup>	Product number	Cis/Trans ratio	Time (h)	Yield <sup>b</sup> (%)
1		<b>2a</b>	56/44	3	75 <sup>c</sup>
2		<b>5a</b>	-	3.5	78
3		<b>2b</b>	55/45	4	81 <sup>c</sup>
4		<b>5b</b>	-	5	76
5		<b>2c</b>	53/47	5	75 <sup>c</sup>
6		<b>5c</b>	-	5.5	78
7		<b>2d</b>	52/48	6	73 <sup>c</sup>

under optimized conditions. These results show that primary and secondary alcohols were selectively reacted via the present method and led to the related diesters of maleate and phthalates in good yields. In the case of the reaction of maleic anhydride with alcohols, two *cis* and

*trans* isomers of maleate diester products were isolated and identified from spectroscopic data (entries 1, 3, 5, 7, 9, 11, Table 1). The esterification of maleic and phthalic anhydrides with alcohols was also tested in the absence of any ionic liquid. The conversion in the presence of IL-OPPh<sub>2</sub>

**Table 1** continued

Entry	Product <sup>a</sup>	Product number	Cis/Trans ratio	Time (h)	Yield <sup>b</sup> (%)
8		<b>5d</b>	-	6.5	75
9		<b>2e</b>	55/45	6	72 <sup>c</sup>
10		<b>5e</b>	-	6	74
11		<b>2f</b>	60/40	5.5	78 <sup>c</sup>
12		<b>5f</b>	-	6	80
13		<b>2g</b>	52/48	13	82 <sup>c</sup>
14		<b>5g</b>	-	14	85

<sup>a</sup> Two *cis* and *trans* diesters of maleate were isolated

<sup>b</sup> Isolated yield

<sup>c</sup> Total yield of *cis* and *trans* isomers

was very higher than that of blank experiment. Therefore, this ionic liquid shows strong catalytic activity.

The recyclability of ionic liquid IL-OPPh<sub>2</sub> in this procedure was examined. The reactions for synthesis of diesters (entries 1 and 2, Table 1) were selected as typical experiments. The ionic liquid was isolated from the reaction mixture and was recycled in the next run without much purification. The results showed that IL-OPPh<sub>2</sub> could be recycled three times without significant loss of its activity.

For comparison, other ionic liquids such as 1-butyl-3-methylimidazolium tetrafluoroborate, [bmim]BF<sub>4</sub>, tetrabutylphosphoniumbromide [PBU<sub>4</sub>]Br and 1-butyl-3-methylimidazolium hexafluorophosphate, [bmim]PF<sub>6</sub> were used for the preparation of maleate and phthalate diesters under above-described optimized conditions. As shown in Table 2, coumarins were synthesized in higher yields and in shorter reaction times using IL-OPPh<sub>2</sub> in comparison with other IL's.

**Table 2** Synthesis of the coumarins using [bmim]BF<sub>4</sub>, [PBU<sub>4</sub>]Br, [bmim]PF<sub>6</sub> and IL-OPPh<sub>2</sub>

Product	Yield <sup>a</sup> (%)					Time (h)		
	[bmim]BF <sub>4</sub>	[PBU <sub>4</sub> ]Br	[bmim]PF <sub>6</sub>	IL-OPPh <sub>2</sub>	[bmim]BF <sub>4</sub>	[PBU <sub>4</sub> ]Br	[bmim]PF <sub>6</sub>	IL-OPPh <sub>2</sub>
<b>2a</b>	48 <sup>b</sup>	52 <sup>b</sup>	48	75 <sup>b</sup>	12	10	9 <sup>b</sup>	3
<b>5a</b>	47	53	50	78	13	11	10	3.5
<b>2b</b>	45 <sup>b</sup>	50 <sup>b</sup>	51	81 <sup>b</sup>	10	12	11 <sup>b</sup>	4
<b>5b</b>	43	51	50	76	11	13	12	5
<b>2c</b>	43 <sup>b</sup>	50 <sup>b</sup>	49	75 <sup>b</sup>	12	9	8 <sup>b</sup>	5
<b>5c</b>	50	48	47	78	13	10	9	5.5
<b>2d</b>	46 <sup>b</sup>	49 <sup>b</sup>	46	73 <sup>b</sup>	14	8	9 <sup>b</sup>	6
<b>5d</b>	44	53	48	75	12	11	10	6.5
<b>2e</b>	43 <sup>b</sup>	46 <sup>b</sup>	51	72 <sup>b</sup>	13	13	11 <sup>b</sup>	6
<b>5e</b>	43	50	50	74	12	12	9	6
<b>2f</b>	45 <sup>b</sup>	49 <sup>b</sup>	49	78 <sup>b</sup>	14	15	8 <sup>b</sup>	5.5
<b>5f</b>	47	52	48	80	12	14	11	6
<b>2g</b>	45 <sup>b</sup>	54 <sup>b</sup>	47	82 <sup>b</sup>	12	11	12 <sup>b</sup>	13
<b>5g</b>	46	52	46	85	11	10	10	14

<sup>a</sup> Yields of isolated product

<sup>b</sup> Total yields of cis and trans isomers

In conclusion, the well-known acid catalyzed esterification reaction was accomplished using phosphinite ionic liquid in the absence of any catalyst and solvent. The diphenylphosphine-functionalized ionic liquid effectively catalyzed the esterification of primary and secondary alcohols with maleic and phthalic anhydrides for the synthesis of symmetrical diesters of maleate and phthalate. Phenols and tertiary alcohols were not reacted with maleic and phthalic anhydrides under these conditions. The advantages of the present protocol are the selective esterification of secondary and primary alcohols in solvent-free conditions, good yields and recyclability of ionic liquid.

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