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# Iron(III)-catalyzed direct synthesis of diphenylmethyl esters from 2-diphenylmethoxypyridine

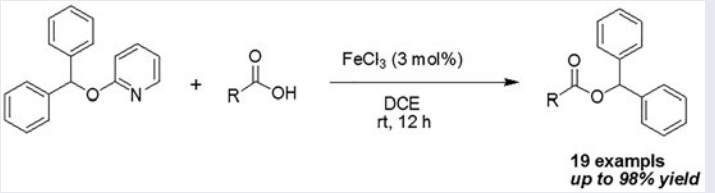
Van Hieu Tran[a,b], Minh Thanh La[a,b], and Hee-Kwon Kim[a,b]

[a]Department of Nuclear Medicine, Molecular Imaging and Therapeutic Medicine Research Center, Chonbuk National University Medical School and Hospital, Jeonju 54907, Republic of Korea; [b]Research Institute of Clinical Medicine of Chonbuk, National University-Biomedical Research Institute of Chonbuk National University Hospital, Jeonju 54907, Republic of Korea

**ABSTRACT**

A highly efficient method has been developed for the synthesis of diphenylmethyl (DPM) esters from 2-diphenylmethoxypyridine. Various carboxylic acids readily reacted with 2-diphenylmethoxypyridine in the presence of $FeCl_3$ as a catalyst to provide the desired DPM esters with high yields. The procedure is facile and enables effective synthesis of a variety of esters for the protection of carboxylic acids.

**GRAPHICAL ABSTRACT**

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**KEYWORDS**
Diphenylmethyl esters; protection; catalysts; iron(III) chloride

## Introduction

Protection and deprotection are commonly used in multistep reactions. As the total synthesis of natural products or bioactive compounds possessing various reactive functional groups has increased, many types of protecting groups have been developed. The diphenylmethyl (DPM) group, first employed for thiol protection,[1] is one of the most common protecting motifs for carboxylic acids because its deprotection can be easily performed under mild conditions. For example, the DPM group can be cleaved from DPM esters by means of acidic treatment or hydrogenation with Pd.[2–8] Due to flexibility in the cleavage of the DPM group, many procedures have utilized DPM esters as intermediates in multistep syntheses of biological compounds, including leukotriene antagonists, adenosine receptor antagonists, and antibiotics.[9–11]

CONTACT Hee-Kwon Kim hkkim717@jbnu.ac.kr Department of Nuclear Medicine, Molecular Imaging and Therapeutic Medicine Research Center, Chonbuk National University Medical School and Hospital, Jeonju 54907, Republic of Korea; Research Institute of Clinical Medicine of Chonbuk, National University-Biomedical Research Institute of Chonbuk National University Hospital, Jeonju 54907, Republic of Korea

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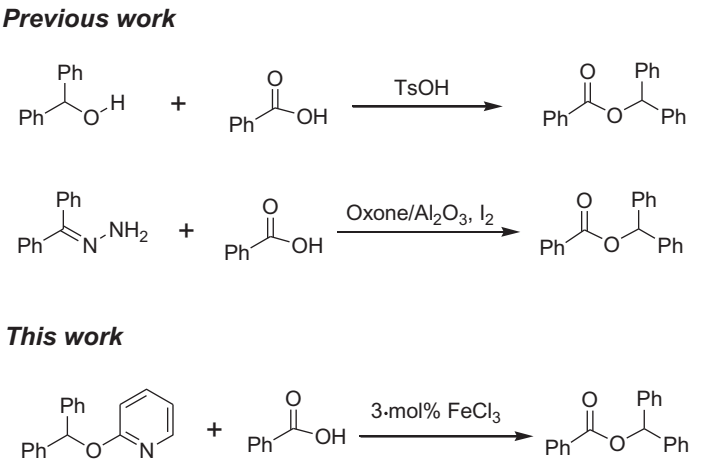
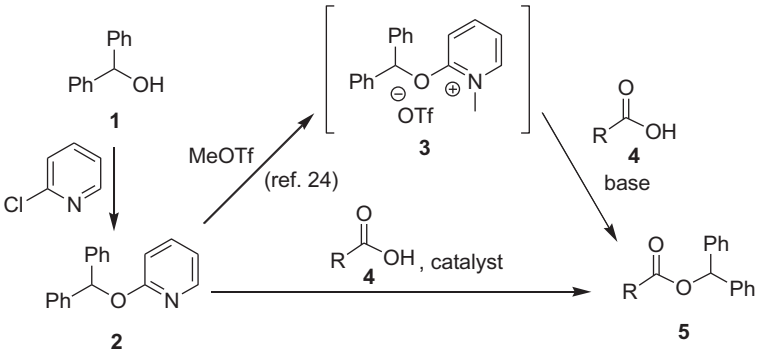



**Scheme 1.** Synthesis of diphenylmethyl esters.

Traditionally, insertion of a DPM group into carboxylic acid to produce the corresponding ester has been carried out by reactions with benzhydrol in the presence of acid (Scheme 1).[12] Such reactions have employed harsh conditions such as the use of strong acids or large amounts of acid, which may affect several sensitive functional groups. Tridiphenylmethyl phosphate, diphenylmethyl diphenyl phosphate, and diphenyldiazo methane have also been used as mild alkylating agents for the protection of carboxylic acids.[13–15] However, the preparation of alkyl phosphates is costly and diazo is a safety risk, specifically having a risk of explosion and being difficult to handle. Instead, benzophenone hydrazone has been utilized as a diazo precursor for the DPM protection of esters. One-pot two-step reactions using benzophenone hydrazine have been used to protect carboxylic acids with oxidative agents such as $I_2$, $PhI(OAc)_2$, oxone, peracetic acid, NaClO with TEMPO catalyst, oxalyl chloride with DMSO, or $MnO_2$.[16–22]

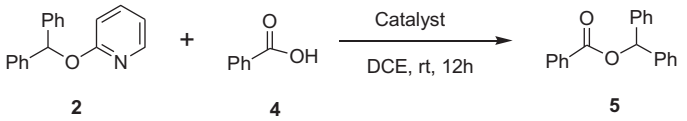
In additions, diphenylmethyl esters were prepared from diphenylmethyl trichloroacetimidate.[23]

2-*O*-substituted pyridines have recently been noticed for the preparation of esters.[24,25] Dudley and colleagues employed 2-benzyloxy-1-methylpyridinium salt as a benzyl transfer agent for syntheses of benzyl esters.[24,25] However, the protocol required two steps and one day of reaction time. In addition, reactions to synthesize benzyl esters required high temperatures such as 83 °C. From the 2-benzyloxy-1-methylpyridine mediated reaction, we expected that this strategy could be extended to other reactions, including the synthesis of DPM esters. We showed DPM esterification using $BF_3{\cdot}OEt_2$ in the previous study.[26] In this study, we tried to find more efficient DPM esterification using a novel metal catalyst. We hypothesized that the combination of 2-diphenyloxy-1-methylpyridine as a starting material and a novel catalyst could lead to highly efficient reaction conditions to form DPM esters. To the best of our knowledge, there is no synthetic protocol using metal-based catalysts to prepare DPM esters from 2-diphenylmethoxypyridine. Herein, we describe a novel facile synthesis of DPM esters from 2-diphenylmethoxypyridine using a catalytic amount of metal-based reagents.




**Scheme 2.** Novel synthetic strategy of diphenylmethyl esters.

**Table 1.** Catalyst screening for DPM esterification.[a]




| Entry | Catalyst | Time | Temp. | Yield[b] (%) |
|---|---|---|---|---|
| 1 | $ZnCl_2$ | 12 | r.t. | NR[c] |
| 2 | $BiCl_3$ | 12 | r.t. | NR[c] |
| 3 | $CuCl_2$ | 12 | r.t. | 2 |
| 4 | $AlMe_3$ | 12 | r.t. | 2 |
| 5 | MnCl | 12 | r.t. | 3 |
| 6 | $ZrCl_4$ | 12 | r.t. | 3 |
| 7 | $FeCl_3$ | 12 | r.t. | 94 |
| 8 | None | 12 | r.t. | NR[c] |

[a]Reaction conditions: compound **2** (1.5 mmol), carboxylic acid (1.0 mmol), catalyst (0.03 mmol), DCE (2 mL), 12 h.
[b]Isolated yield after purification via flash column chromatography.
[c]No reaction.

## Result and discussion

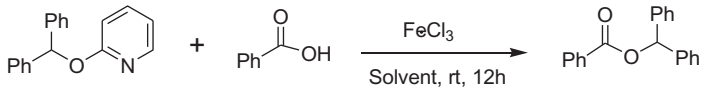
DPM esters were synthesized from diphenylmethyl alcohol in 2-step routes, as shown in Scheme 2. In the first reaction, 2-diphenylmethoxypyridine (compound **2**) was readily obtained by treating diphenylmethyl alcohol with 2-chloropyridine in the presence of potassium and 18-crown-6 according to our previously reported method.[26]

Recently we noticed that the previous method using MeOTf to produce 2-benzyloxy-1-methylpyridinium salt, followed by carboxylic acid treatment, provided DPM esters in low yields. We assume that a catalytic reagent such as a Lewis acid could be a better choice in DPM esterification and etherification.

In the initial study, benzoic acid was chosen as a substrate for the reaction with 2-diphenylmethoxypyridine. Esterification reactions of benzoic acid were carried out in the presence of 1.5 equiv. of 2-diphenylmethoxypyridine, 1.0 equiv. of benzoic acid, and 0.03 equiv. of various Lewis acids at room temperature for 12 h, and the reaction yields of the corresponding esters were examined.

The screening study indicated that reactions with Lewis acids such as $ZnCl_2$ and $BiCl_3$ could not produce the target product (Table 1), and that a series of Lewis acids, including $CuCl_2$, $AlMe_3$, $MnCl_2$, and $ZrCl_4$, could produce only small amounts of the

**Table 2.** Solvent screening for in situ esterification.[a]




2 4 5

| Entry | Catalyst (equiv.) | Solvent | Temp. | Yield[b] (%) |
|---|---|---|---|---|
| 1 | $FeCl_3$ (0.03) | MeCN | r.t. | 26 |
| 2 | $FeCl_3$ (0.03) | THF | r.t | 22 |
| 3 | $FeCl_3$ (0.03) | Toluene | r.t. | 61 |
| 4 | $FeCl_3$ (0.03) | 1,4-dioxane | r.t. | NR[c] |
| 5 | $FeCl_3$ (0.03) | $CH_2Cl_2$ | r.t. | 73 |
| 6 | $FeCl_3$ (0.03) | DCE | r.t. | 94 |
| 7 | $FeCl_3$ (0.2) | DCE | r.t. | 95 |
| 8 | $FeCl_3$ (0.1) | DCE | r.t. | 94 |
| 9 | $FeCl_3$ (0.01) | DCE | r.t. | 74 |
| 10[d)] | $FeCl_3$ (0.03) | DCE | 50 °C | 95 |
| 11[d)] | $FeCl_3$ (0.03) | DCE | 70 °C | 95 |

[a]Reaction conditions: compound **2** (1.5 mmol), carboxylic acid (1.0 mmol), solvent (2 mL), 12 h.
[b]Isolated yield after purification via flash column chromatography.[c]No reaction.
[c]Reaction was conducted for 4 h.

desired DPM esters: most reaction yields were less than 10%. Finally, $FeCl_3$ was tested for the synthesis of DPM esters; the reaction in the presence of $FeCl_3$ yielded the desired esters with significantly enhanced yield (94%). This result means that $FeCl_3$ could be used as an effective catalytic reagent for DPM esterification.

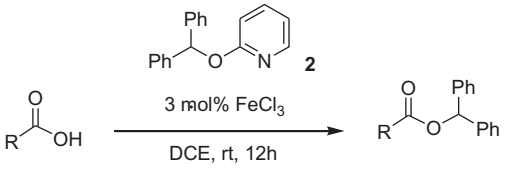
Next, the effect of the solvent upon reaction was explored to determine the optimal reaction conditions. As shown in Table 2, reactions using $FeCl_3$ in THF, MeCN, and 1,4-dioxane provided poor yields in the production of DPM ester. Using toluene as the reaction solvent increased the transformation yield of carboxylic acid to the target product to 61%, but many unreacted starting materials were still observed. The chlorinated solvents including dichloromethane (DCM) and dichloroethane (DCE) were also tested for DPM esterification and fortunately gave significantly enhanced reaction yields: the target DPM ester was obtained in 73% yield from reaction in DCM and in 94% yield in DCE. Thus, DCE was chosen as the most effective solvent for further study of $FeCl_3$-catalyzed DPM esterification reactions.

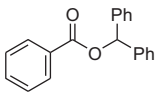
In addition, a series of amounts of $FeCl_3$ (0.2 equiv., 0.1 equiv., 0.03 equiv., and 0.01 equiv.) was tested for DPM esterification. As listed in Table 2, the synthetic yield for the desired ester increased with an increasing amount of $FeCl_3$ to 0.03 equiv. (94% at room temperature in DCE), though further addition of $FeCl_3$ did not improve the reaction yield. These results suggest that efficient preparation of DPM ester compounds can be achieved using 0.03 equiv. (3 mol%) of $FeCl_3$ catalyst.

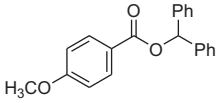
Temperature effect on DPM esterification was also investigated. The target product was prepared with yields of 95% at 50 °C and 70 °C for 4 h. But, we selected room temperature for future study because mild reaction condition is more suitable.

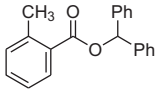
Utilizing these optimized reaction conditions, the scope of DPM ester synthesis was explored. These reactions of carboxylic acids with 2-diphenylmethoxypyridine were carried out using 3 mol% of $FeCl_3$ in DCE at room temperature for 12 h. The synthesis of DPM esters was demonstrated to be tolerant to various carboxylic acid starting

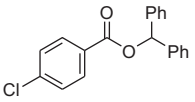
**Table 3.** Scope of DPM esterification using 2-diphenylmethoxypyridine.[a]

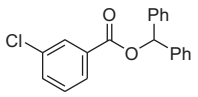


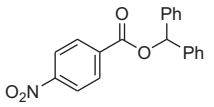


**5a**, 94%

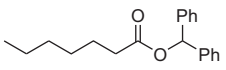

**5b**, 93%

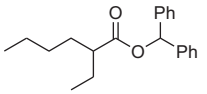

**5c**, 92%

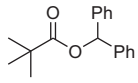

**5d**, 75%

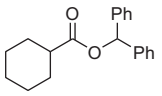

**5e**, 92%

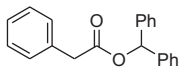

**5f**, 84%

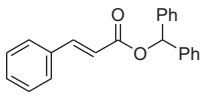

**5g**, 92%

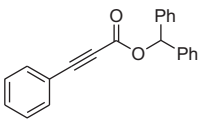

**5h**, 84%

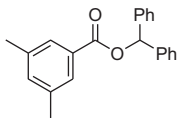

**5i**, 93%

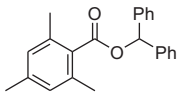

**5j**, 95%

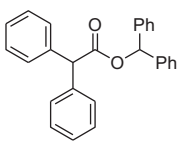

**5k**, 93%

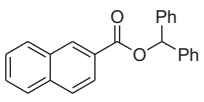

**5l**, 98%

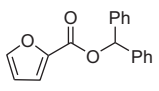

**5m**, 97%

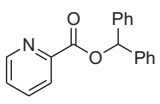

**5n**, 96%


**5o**, 93%

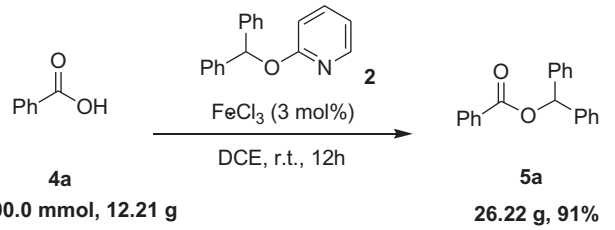

**5p**, 95%


**5q**, 94%


**5r**, 93%


**5s**, 83%

[a]Reaction conditions: carboxylic acid (1.0 mmol), compound **2** (1.5 mmol), $FeCl_3$ (0.03 mmol), DCE (2 mL), r.t. for 12 h.




**Scheme 3.** Gram-scale reaction of benzoic acid (**4a**) with 2-diphenylmethoxypyridine (**2**).

materials, as listed in Table 3. Several substituted aromatic carboxylic acids with different electronic effects were tested to synthesize DPM esters. Reactions of benzoic acids with electron-donating groups (methoxy and methyl) and electron-withdrawing substituents (nitro and chloro) resulted in successful conversions of carboxylic acids to the desired DPM esters in the range from 75% to 93% yield (**5b–5f**). This result suggests that the synthetic yields of DPM esterification using $FeCl_3$ were not significantly influenced by the differences in electronic effects arising from these substituents of benzoic acids.

Alkyl-substituted carboxylic acids were also used to prepare DPM esters. The esterification of alkyl-substituted carboxylic acids afforded the desired products in high yields (**5g–5k**). Particularly, pivalic acid which has *tert*-butyl group, and cyclohexane carboxylic acid which is a cyclic substrate also reacted readily with 2-diphenylmethoxypyridine to provide the corresponding esters in yields of 93% and 95%, respectively.

Alkenyl- and alkynyl-substituted carboxylic acids, which are unsaturated carboxylic acids with reactive $\pi$ bonds, were also employed in DPM esterification to assess the scope of this method. These substrates were transformed into the desired esters in excellent yields without the formation of side products (**5l** and **5m**).

The scope of this synthetic protocol was extended to carboxylic acids with more bulky groups that could have steric effects on the reactions. Treatment of 3,5-dimethylbenzoic acid and 2,4,6-trimethylbenzoic acid in the presence of catalytic $FeCl_3$ led to efficient synthesis of the desired product with yields of 96% and 93%, respectively (**5n** and **5o**). Furthermore, diphenylacetic acid, which has two phenyl groups and thus potentially a large steric effect, provided the target product **5p** in 95% yield under the same conditions.

In addition, $FeCl_3$ was used with heterocyclic acid compounds to examine whether it also catalyzed these compounds. In these experiments, 2-furoic acid bearing oxygen, and 2-picolinic acid bearing nitrogen were treated with 2-diphenylmethoxypyridine, providing the corresponding DPM esters in yields of 93% and 83%, respectively (**5r** and **5s**). This indicated that $FeCl_3$ is an effective catalytic reagent for the successful synthesis of DPM esters from heterocyclic carboxylic acids under mild conditions.

Next, a scale-up of DPM esterification was conducted (Scheme 3). The DPM ester was successfully prepared in a gram-scale reaction of benzoic acid **4a** (100.0 mmol, 12.21 g) with 2-diphenylmethoxypyridine **2**; the target ester product **5a** was produced in 91% yield via the optimized reaction conditions. This reaction was thus demonstrated to be effective and scalable.

## Conclusion

In conclusion, a novel reaction method was developed for practical DPM esterification of various carboxylic acids. Novel synthetic methods of DPM esters are important to develop protecting procedures for organic chemistry and to prepare bioactive compounds. This study featured $FeCl_3$-catalyzed synthesis of carboxylic acids with 2-diphenylmethoxypyridine to produce DPM esters with high yield. The novel procedures are highly efficient in yielding DPM esters, and thus have potential application to the preparation of bioactive compounds such as drugs.

## Experimental

Reagents and solvents were commercially available and used as received. Column chromatography was performed using silica gel 60 (0.040–0.063 mm) and eluted with proper mixture (DCM/hexane). All of the new compounds were identified by 400 MHz $^1H$ and 100 MHz $^{13}C$ NMR spectra in deuterated chloroform ($CDCl_3$) with tetramethylsilane (TMS) as an internal reference, and high resolution mass spectroscopy (Supplementary Data). The identity of the known compounds was established by the comparison of their $^1H$ and $^{13}C$ NMR peaks with the authentic values.

### *General procedure for the preparation of DPM ester compounds (5a–5s)*

To a solution of benzoic acid (0.122 g. 1.00 mmol) and 2-diphenylmethoxypyridine (0.39 g, 1.50 mmol) in DCE (2 mL) $FeCl_3$ (0.0048 g. 0.03 mmol) was added. The mixture was stirred at room temperature for 12 h. The reaction mixture was extracted with ethyl acetate (2 × 10 mL), and then washed with water (10 mL), followed by brine (10 mL). The organic layer was dried over anhydrous sodium sulfate and concentrated under reduced pressure. The resulting residue was then purified using flash column chromatography on silica gel with hexane-$CH_2Cl_2$ as eluent to afford the desired product **5a** as a white solid (0.271 g, 94%).

### *Selected spectroscopic data*

#### *Benzydryl benzoate (5a)*

White solid (94% yield); m.p. 90–92 °C; $^1H$ NMR (600 MHz, $CDCl_3$) $\delta$ 8.15 (d, $J = 6.0$ Hz, 2H), 7.57 (td, $J = 6.5$ Hz, $J = 1.5$ Hz, 1H), 7.50–7.44 (m, 6H), 7.36 (t, $J = 6.0$ Hz, 4H), 7.29 (t, $J = 6.0$ Hz, 2H), 7.13 (s, 1H); $^{13}C$ NMR (150 MHz, $CDCl_3$) $\delta$ 165.7, 140.4, 133.3, 130.3, 129.9, 128.7, 128.6 128.1 127.2, 77.5; HRMS (ESI) *m/z* $(M + H)^+$ calcd for $C_{20}H_{17}O_2$ = 289.1229, found 289.1227.

#### *Benzhydryl 4-chlorobenzoate (5d)*

White solid (75%); m.p. 90–92 °C; $^1H$ NMR (400 MHz, $CDCl_3$) $\delta$ 8.10 (dt, $J = 8.8$ Hz, $J = 2{,}4$ Hz, 2H), 8.48–7.31 (m, 12H), 7.14 (s, 1H); $^{13}C$ NMR (100 MHz, $CDCl_3$) $\delta$

164.74, 140.02, 139.63, 131.17 (2C), 128.81 (2C),128.61(4C), 128.18 (2C), 127.12 (4C); HRMS (ESI) *m/z* $(M+H)^+$ calcd for $C_{20}H_{16}ClO_2$ = 323.0839, found 323.0840.

### *Benzhydryl heptanoate (5g)*

Colorless oil (92%); $^1$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.38–7.31 (m, 10H), 6.93 (s, 1H), 2.47 (t, $J$ = 7.6 Hz, 2H), 1.69 (m, 2H), 1.30 (m, 6H), 0.91 (m, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 172.81, 140.38 (2C), 128.48 (4C), 127.84 (2C), 127.09 (4C), 76.60, 34.61, 31.46, 28.79, 24.94, 22.49, 14.04; HRMS (ESI) m/z $(M+H)^+$ calcd for $C_{20}H_{25}O_2$ = 297.1855, found 297.1856.

### *Benzhydryl 2-phenylacetate (5k)*

White solid (93%); m.p. 45–47 °C; $^1$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.34–7.30 (m, 15H), 6.91 (s, 1H), 3.77 (s, 2H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 170.47, 140.10, 133.83, 129.40, 128.59, 128.48, 127.89, 127.15, 127.02. 77.26, 41.67; HRMS (ESI) *m/z* $(M+H)^+$ calcd for $C_{21}H_{19}O_2$ = 303.1385, found 303.1387.

### *Benzhydryl furan-2-carboxylate (5r)*

White solid (93%); m.p. 89–91 °C; $^1$H NMR (400 MHz, $CDCl_3$) $\delta$ 7.63–7.62 (m, 1H), 7.46–7.32 (m, 11H), 7.14 (s, 1H), 6.55–6.54 (m, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) $\delta$ 157.75, 146.57 (2C), 144.64, 139.90, 128.56 (4C), 128.05 (2C), 127.21 (4C), 118.35, 111.89, 77.19; HRMS (ESI) *m/z* $(M+H)^+$ calcd for $C_{18}H_{15}O_3$ = 279.1021, found 279.1024.

## Disclosure statement

No potential conflict of interest was reported by the authors.

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