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Organic hydrogen phosphites and hydrogen phosphates catalyzed Friedel–Crafts amidoalkylation of indoles with aryl aldimines

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1. Introduction

The 3-substituted indole structural motifs are widely presented in pharmaceuticals, agrochemicals, indole alkaloids and synthetic indole derivatives. In the past 10 years, synthesis of 3-indolyl derivatives has attracted lot of interest and particularly synthesis of biologically active 3-indolyl methanamines.¹ Correspondingly, the Friedel-Crafts alkylation² of indoles with imines has turned out to be an atom economic and short-armed method for the preparation of 3-indolyl methanamine derivatives via addition of sp² C-H bonds of indoles to C=N double bond of imines. Literatures have been reported that various lewis acids, such as TiCl₄,³ Sc(OTf),⁴ Dy(OTf)₃,⁵ InCl₃,⁶ SmI₃,⁷ AuCl₃/AgOTf,⁸ BF₃·OEt₂,⁹ are employed to catalyze the Friedel-Crafts amidoalkylation of indole with imines. However, due to the intrinsic instability of the benzylic amine derivatives under the acidic reaction conditions. most of the reactions catalyzed by lewis acids mentioned above furnish 3-indolyl methanamine derivatives, and simultaneously generate the bisand tris(indolyl)methanes (BIMs and TIMs)^{5,10} in considerable amounts (Scheme 1). Meanwhile, some strategies, such as utilizing of ionic liquids,¹⁰ aqueous phase,¹¹ ultrasound¹² and Microwave,¹³ as well as mild reagents such as montmorillonite clay K-10/KSF¹⁴ and exchange resins,¹⁵ have been adopted and employed for the corresponding Friedel-Crafts alkylation reactions, which provide 3-indolyl methanamines and also accompany with the BIMs and TIMs as by-products or main products in some cases.

ABSTRACT

A highly efficient and selective Friedel–Crafts amidoalkylation reaction of indoles with N-Ts aryl aldimines has been developed utilizing dimethyl hydrogen phosphite or diphenyl hydrogen phosphate as the organocatalysts, providing a facile and cost-effective process for synthesis of 3-indolyl methanamine derivatives in good to excellent yields. This transformation displays a broad substrate scope and wide functional-group tolerability, regardless of the electronic and steric properties of N-Ts aryl aldimines. Given that the developed catalytic Friedel–Crafts amidoalkylation reaction exhibits several salient features such as metal-free catalysis, high efficiency, low cost and mild reaction condition, this process might have practical applications in the synthesis of 3-indolyl methanamine derivatives.

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Just in recent years, the Friedel–Crafts amidoalkylation of indoles with different imines have been successfully developed, including asymmetric and non-asymmetric versions catalyzed by metal and/or metal-free catalysts, thus allowing facile access to 3-indolyl methanamine derivatives in good yields or with high enantioselectivities.¹⁶ Especially, some soft brønsted acids turn out to be effective catalysts for the Friedel–Crafts amidoalkylation of indoles with imines. You, Terada, and Antilla groups, respectively, have demonstrated that chiral binaphthyl derived phosphoric acids can catalyze the asymmetric Friedel–Crafts alkylation of indoles with imines, affording the corresponding 3-indolyl methanamine derivatives in high yields with excellent enantioselectivities.^{16e-g} Kobayashi and Shirakawa have disclosed that the linear alkyl carboxylic acids are effective catalysts for three-component aza-Friedel–Crafts reactions in water, providing 3-indolyl



Scheme 1. Friedel–Crafts amidoalkylation of indoles with N-Ts aryl aldimines catalyzed by Lewis acid and brønsted acids.

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Table 1



Entry	Catalyst		4a Yield ^c (%)	5 Yield ^c (%)
1	O MeO-P-H OMe	1a	86	<5
2	EtO-P-H OEt	1b	42	12
3	O II Pri O-P-H O <i>i</i> Pr	1c	34	10
4	O PhO-P-H OPh	1d	65	8
5	O Ph-P-H Ph	1e	_	_
6 ^d	O II PhO-P-OH OPh	1f	83	<5
7	O Ph-P-OH H	1g	36	60
8	O H Ph-P-OH OH	1h	27	69
9 ^d	(R)-BINOL phosphoric acid	1i	80	<5

^a The photographs: (i) before reaction and (ii) after reaction for entry 1.

^b Reaction conditions: catalysts **1a**-**i** (20 mol %), **2a** (1.5 mmol), **3a** (0.5 mmol) in toluene (3.0 mL) at rt for 2 h.

^c Isolated yield.

^d Reaction conditions: catalyst **1f** (20 mol %), **2a** (2.5 mmol), **3a** (0.5 mmol), toluene (3.0 mL) at rt for 2 h.

methanamines in high yields.¹⁷ Herein, we would like to present our preliminary results on the Friedel–Crafts amidoalkylation of indoles with N-Ts aryl aldimines catalyzed by organic hydrogen phosphites or hydrogen phosphates, providing the 3-indolyl methanamines in high yields.

2. Results and discussion

Initially, several common hydrogen phosphites or hydrogen phosphates **1a–i** were examined for the catalysis of the Friedel– Crafts amidoalkylation of indole **2a** and N-Ts aldimine **3a** with 20 mol % of the catalyst loading in toluene at room temperature. The results are summarized in Table 1. We found that dimethyl hydrogen phosphite **1a** was quite effective catalyst for the reaction of **3a** with one equivalent amount of **2a**, affording the desired product **4a** in 50% yield within 2 h, albeit with bisindole **5** in 13% yield as by-products. When two equivalent amounts of indole **2a** were used, the reaction did not proceed faster but led to the desired

product in a slightly higher yield (68%). When increasing the amount of indole 2a to 3 equiv, an optimal result was obtained in terms of 86% yield with bisindole 5 in less than 5% yield. Subsequently, 5 equiv of indoles were introduced and the yield was not obviously affected (87%). Then diethyl hydrogen phosphite 1b and diisopropyl hydrogen phosphite 1c were examined for this reaction, affording the desired products in 42% and 34% yields and the by-products of bis(indolyl)methanes in 12% and 10% yields, respectively (entries 2–3). In comparison with **1b** and **1c**, diphenyl hydrogen phosphite **1d** was amenable to generate the desired product in 65% yield and by-products in 8% yield (entry 4). Unexpectedly, diphenylphosphine oxide 1e was found to be ineffective for the reaction, where essentially no product was obtained after prolonged the reaction time (entry 5). To our delight, diphenyl hydrogen phosphate **1f** could smoothly catalyze this reaction to provide the desired product in 78% yield, when 3 equiv amounts of 2a were utilized. For this case, increasing the amount of 2a relative to that of **3a** was found to slightly improve the yield of **4a**. When 5 equiv of 2a were used, the desired product was obtained in 83% yields, which was accompanied with less than 5% yield of the by-products (entry 6). Phenyl phosphinic acid 1g, which was confirmed as effective catalyst for the acylcyanation of aldimines and acylcyanides by List and Pan,¹⁸ was incompetent for the reaction to provide the desired product 4a in 36% yield with 60% yield of undesired by-products 5 being formed (entry 7). In addition, phenylphosphonic acid **1h** was also inefficient for this transformation, providing the desired product only in 27% yield and the undesired by-products 5 in 69% yield (entry 8). Moreover, having established by You's group,^{16e} the binaphthyl scaffold phosphoric acid **1i** was the really effective catalyst for this reaction, giving the desired product in 80% yield (entry 9, Table 1). Thus, having critically examined the mentioned catalysts, we found that the dimethyl hydrogen phosphite 1a and diphenyl hydrogen phosphate 1f were effective catalysts. Finally, it should be worth noting that the reactions catalyzed by 1a and 1f have proceeded very fast. The reaction mixture generally becomes cloudy and thick within 10 min (picture (i) and (ii) for entry 1 in Table 1).

Further results on the optimization of the other parameters for the reaction of indole **2a** and aldimine **3a** catalyzed by dimethyl hydrogen phosphite **1a** and diphenyl hydrogen phosphate **1f**, including solvent and catalyst loading, are summarized in Table 2. After screening several common solvents for the reaction, the results indicated that toluene was the optimal solvent for the reaction, affording the desired product in the highest yield (entries 1– 7). Furthermore, when the catalyst loading was decreased to 15 mol %, the yield was not influenced too much. When the cata-

Table 2			
Optimization	of reaction	conditions ^a	

Entry	Catalyst (mol %)	Solvent	Yield ^b (%)
1	1a (20)	Toluene	86
2	1a (20)	Xylene	70
3	1a (20)	Benzene	65
4	1a (20)	CHCl ₃	54
5	1a (20)	DCM	76
6	1a (20)	ClCH ₂ CH ₂ Cl	69
7	1a (20)	THF	<5
8	1a (15)	Toluene	90
9	1a (10)	Toluene	51
10	1a (5)	Toluene	46
11 ^c	1f (15)	Toluene	81
12 ^c	1f (10)	Toluene	85
13 ^c	1f (5)	Toluene	75

 $^{\rm a}~$ Reaction conditions: **2a** (1.5 mmol), **3a** (0.5 mmol), solvent (3.0 mL) at rt for 2 h. $^{\rm b}~$ Isolated yields.

^c Reaction conditions: **2a** (2.5 mmol), **3a** (0.5 mmol), toluene (3.0 mL) at rt for 2 h.

2

Table 3

Dimethyl hydrogen phosphite **1a** catalyzed the Friedel–Crafts amidoalkylation of indoles with N-Ts aryl aldimines^a



Entry	Indole (R)	Aldimine (Ar)	Product	Yield ^b (%)
1	H (2a)	Ph (3a)	4a	90
2	H (2a)	p-FC ₆ H ₄ (3b)	4b	89
3	H (2a)	p-ClC ₆ H ₄ (3c)	4c	91
4	H (2a)	p-BrC ₆ H ₄ (3d)	4d	82
5	H (2a)	p-MeOC ₆ H ₄ (3e)	4e	80
6	H (2a)	$p-CF_{3}C_{6}H_{4}$ (3f)	4f	88
7	H (2a)	p-MeC ₆ H ₄ (3g)	4g	83
8	H (2a)	o-FC ₆ H ₄ (3h)	4h	91
9	H (2a)	o-ClC ₆ H ₄ (3i)	4i	88
10	H (2a)	o-BrC ₆ H ₄ (3j)	4j	88
11	H (2a)	o-MeOC ₆ H ₄ (3k)	4k	81
12	H (2a)	2,4-Cl ₂ C ₆ H ₃ (31)	41	84
13	H (2a)	$m-MeC_{6}H_{4}(3m)$	4m	85
14	H (2a)	$m-ClC_{6}H_{4}(3n)$	4n	89
15	H (2a)	thiophen-2-yl (30)	4o	60
16	H (2a)	1-Naphthyl (3p)	4p	84
17	H (2a)	2-Naphthyl (3q)	4q	81
18	5-Me (2b)	Ph (3a)	4r	57
19	5-Br (2c)	Ph (3a)	4s	59
20	7-Me (2d)	Ph (3a)	4t	54

^a Reaction conditions: **2a-d** (1.5 mmol), **3a-q** (0.5 mmol) in toluene (3.0 mL) at rt for 2 h.

^b Isolated yield.

lyst loading was continued to decrease to 10 mol % and 5 mol %, the yields rapidly dropped to 51% and 46%, respectively (entries 8–10). On the other hand, the catalyst loading was also investigated for the Friedel–Crafts amidoalkylation of indole **2a** with N-Ts aldimine **3a** catalyzed by diphenyl hydrogen phosphate **1f**, and 10 mol % of catalyst loading was found to be suitable for the transformation, affording the desired product in 85% yield (entries 11–13).

Having established the optimal reaction conditions, we proceeded to explore the substrate scope and limitation utilizing dimethyl hydrogen phosphite 1a as the catalyst. As shown in Table 3, the results indicated that all the reactions between N-Ts aryl aldimines **3a-q** and indole derivatives **2a-d** were performed smoothly in the presence of 15 mol % dimethyl hydrogen phosphite 1a. The N-Ts aryl aldimines 3a-n bearing ether electrondonating substituents (-Me, -OMe) or electron-withdrawing substituents (-F, -Cl, -Br, -CF₃) at para-, ortho- or meta-position on phenyl group were all well tolerated, and the electronic and steric properties of N-Ts aryl aldimines displayed little effect on their reactivities, leading to their desired products in 80-91% yields (entries 1-14). Moreover, the substrate 30 bearing 2-thienyl group was also a suitable substrate for this reaction, furnishing the product 4o in 60% yield (entry 15), Furthermore, aldimines 3p and 3q bearing α -naphthyl, β -naphthyl groups also gave the desired products **4p** and **4q** in 84% and 81% yields, respectively (entries 16–17). Among them, the reactions for substrates **3e**, **3g**, **3o** and **3g** occurred relatively slow, as the time for turning translucent was obviously prolonged for those reactions. Unfortunately, the present protocol suffered from limitation in indole derivatives 2b-d as substrates. Under the optimal conditions, the Friedel-Crafts amidoalkylation of indole derivatives **2b-d** with **3a** proceeded sluggishly to afford the desired adducts 4r-t in moderate yields (54-59%) within 2 h (entries 18-20). Further extending the reaction time, the yields of bis(indolyl)methanes (BIMs) were visibly increased.

Table 4

Diphenyl hydrogen phosphate **1f** catalyzed the Friedel–Crafts amidoalkylation of indoles with N-Ts aryl aldimines^a



Entry	Indole (R)	Aldimine (Ar)	Product	Yield ^b (%)
1	H (2a)	Ph (3a)	4a	85
2	H (2a)	p-FC ₆ H ₄ (3b)	4b	80
3	H (2a)	$p-ClC_6H_4$ (3c)	4c	78
4	H (2a)	<i>p</i> -BrC ₆ H ₄ (3d)	4d	79
5	H (2a)	p-MeOC ₆ H ₄ (3e)	4e	75
6	H (2a)	p-CF ₃ C ₆ H ₄ (3f)	4f	81
7	H (2a)	p-MeC ₆ H ₄ (3g)	4g	80
8	H (2a)	o-FC ₆ H ₄ (3h)	4h	80
9	H (2a)	o-ClC ₆ H ₄ (3i)	4i	81
10	H (2a)	o-BrC ₆ H ₄ (3j)	4j	89
11	H (2a)	o-MeOC ₆ H ₄ (3k)	4k	81
12	H (2a)	2,4-Cl ₂ C ₆ H ₃ (31)	41	79
13	H (2a)	m-MeC ₆ H ₄ (3m)	4m	78
14	H (2a)	m-ClC ₆ H ₄ (3n)	4n	81
15	H (2a)	Thiophen-2-yl (30)	4o	75
16	H (2a)	1-Naphthyl (3p)	4p	87
17	H (2a)	2-Naphthyl (3q)	4q	86
18	5-Me (2b)	Ph (3a)	4r	87
19	5-Br (2c)	Ph (3a)	4s	89
20	7-Me (2d)	Ph (3a)	4t	86
21	5-Me (2b)	o-ClC ₆ H ₄ (3i)	4u	95
22	5-Br (2c)	o-ClC ₆ H ₄ (3i)	4v	90
23	7-Me (2d)	o-ClC ₆ H ₄ (3i)	4w	92

^a Reaction conditions: **2a-d** (2.5 mmol), **3a-q** (0.5 mmol) in toluene (3.0 mL) at rt for 2 h.

^b Isolated yield.

The substrate scope and Limitation were also investigated for the Friedel-Crafts amidoalkylation of N-Ts indole derivatives 2a**d** with arvl aldimines **3a–q** catalyzed by diphenyl hydrogen phosphate **1f**. The results are summarized in Table 4, the reactions proceeded more successfully in the presence of 10 mol % of diphenyl hydrogen phosphate 1f at rt in toluene for 2 h. The substrates **3a–n** with different electronic and steric properties all led to their desired products 4a-n in good yields (75-89%) (entries 1-14). N-Ts aryl aldimines **30-q** containing ether naphthyl groups or heteroaromatic ring were all suitable substrates for this transformation, affording the corresponding products 40-q in 75-87% yields (entries 15-17). Pleasingly, the Friedel-Crafts amidoalkylation of indole derivatives 2b-d with N-Ts aryl aldimine 3a and 3i, respectively, performed very well to afford the desired products 4r-w in good to excellent yields (86-95%) (entries 18-23). It should be noted that all the reactions have to be timely quenched once the starting materials disappear, because the amount of bisindole by-products would be enhanced with the prolonged reaction time

In summary, we have disclosed that dimethyl hydrogen phosphite or diphenyl hydrogen phosphate can efficiently catalyze the Friedel–Crafts amidoalkylation of indoles with N-Ts aryl aldimines, selectively affording 3-indolyl methanamines in good to excellent yields. Furthermore, this transformation displays a broad substrate scope and wide functional-group tolerability, regardless of the electronic and steric properties of N-Ts aryl aldimines. The dimethyl hydrogen phosphite ($C_2H_7O_3P$) as the organocatalyst contains two carbons, and the molecular weight is only 110.05, which might be regarded as an atom economic and low carbonic catalyst. More importantly, the present protocol displays several salient features such as metal-free catalysis, high efficiency, low cost and mild reaction condition, which might have practical appli-

cations in the synthesis of 3-indolyl methanamine derivatives. Further application of orthophosphorous acids or phosphoric acids as the catalysts and extension of the reaction scope are currently underway in our laboratory.

2.1. General procedure for the catalytic Friedel–Crafts amidoalkylation

N-Sulfonyl aldimines **3** (0.5 mmol) and dimethyl hydrogen phosphite **1a** (0.075 mmol, 7.0 μ L) or diphenyl hydrogen phosphate **1f** (0.05 mmol, 12.8 mg) were dissolved in toluene (3.0 mL). The solution was stirred at room temperature for 10 minutes. Subsequently, **2** (1.5 or 2.5 mmol) was added in one portion. After the reaction was complete (monitored by TLC), 10% NaHCO₃ (6 mL) was added to quench the reaction. The mixture was extracted with ethyl acetate (3 × 5 mL). The combined organic phase was washed by H₂O (5 mL) and brine (5 mL) and dried over anhydrous Na₂SO₄. After filtration, the solvent was removed under reduced pressure and the residue was purified by flash chromatography (DCM/petroleum ether = 1/3 to 1/0) to afford the product, which was determined by ESI-MS, ¹H and ¹³C NMR spectroscopy.

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Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.tetlet.2011.07.010.

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