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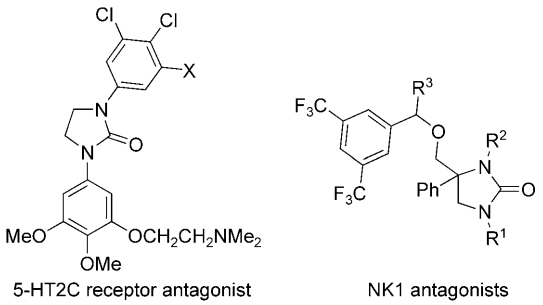
Palladium-Catalyzed Three-Component Reaction of 2,3-Allenyl Amines, Isocyanates, and Organic Halides: A Diversified Assembly of Imidazolidinones

Wei Shu,[a] Qiong Yu,[b] Guochen Jia,*[c] and Shengming Ma*[a, b]

Imidazolidinones are important moieties that are present in various biologically active compounds,[1] such as 5-HT2C receptor antagonist[2] and NK1 antagonists[3] (Scheme 1). In general, imidazolidinones can be prepared by cyclization of α-amino derivatives of ketones, aldehydes, and related compounds,[4] or by derivatization of imidazolidinones.[5] Recently, Shi and co-workers have reported the copper(I)-catalyzed α-amination of aryl ketones and diamination of disubstituted terminal olefins to afford substituted imidazolidinones.[6]



Scheme 1. Some biologically active compounds containing imidazolidinones.

On the other hand, the design of multicomponent reactions[7] for the construction of organic molecules has attracted more and more attention. This strategy offers significant advantages over the classical step-by-step approaches, as it allows the formation of several bonds in just one single synthetic operation. Furthermore, in this way complex molecular architectures can be built from simple precursors without the need for the isolation of intermediates.

During the past decades, palladium-catalyzed two-component cyclization reactions of functionalized allenes with organic halides represented one of the most powerful and versatile tools to construct cyclic compounds.[8] As we know, isocyanates have found a wide range of applications in organic synthesis, particularly in the synthesis of heterocycles.[9] Based on the analysis of the structures of biologically active compounds shown in Scheme 1, we envisioned that these potentially important imidazolidinones may be prepared in a three-component reaction of 2,3-allenyl amines[10] with organic halides in the presence of isocyanates. In this process, the π-allylic palladium moiety would be formed by carbometalation[11] of 2,3-allenyl amines and the urea anion would be formed from the reaction of amines with isocyanates.[12] The C=C bond adds the opportunity to convert it into the required ether functionality through the intermediacies of ketones and alcohols; R^1, R^2, and R^3 from the three starting compounds would provide the diversity required by the bio-screening. The challenge would be the premature cyclization of intermediate *syn*-**M1** forming vinylic azacyclopropanes or *anti*-**M1** forming 2,5-dihydroprroles and the formation of the seven-membered three-component products from *anti*-**M2** (Scheme 2).[8] Herein, we report the realization of such a diversified protocol for the efficient synthesis of imidazolidinones with an excellent selectivity.

We started our effort by reacting 2,3-allenyl amine **1a** with phenyl isocyanate **2a** and iodobenzene **3a** in the presence of K_2CO_3 (2 equiv) catalyzed by [$Pd(PPh_3)_4$] (5 mol%) in THF at 70°C. To our delight, the desired product **4a** was obtained in 72% yield after 22 h in this first try. After screening of the solvent effect, we found that the reaction proceeded smoothly in all the solvents tested, and afforded **4a** in moderate to good yields (Table 1), and CH_3CN gave the best result, thus affording **4a** in 96% yield (Table 1, entry 6). The reaction with [$Pd_2(dba)_3$]·$CHCl_3$ (2.5 mol%; dba = dibenzylideneacetone) and PPh_3 (10 mol%) gave the product **4a** in a slightly lower yield (Table 1, entry 7)

Next we examined the base effect on this reaction. Among the bases tested, potassium carbonate, which was the first to be tested (Table 1), resulted to be the best one (Table 2, entries 1–4). Reducing the amount of potassium

[a] W. Shu, Prof. Dr. S. Ma
State Key Laboratory of Organometallic Chemistry
Shanghai Institute of Organic Chemistry
Chinese Academy of Sciences
354 Fenglin Lu, Shanghai 200032 (P. R. China)
Fax: (+86) 21-62609305
E-mail: masm@sioc.ac.cn

[b] Q. Yu, Prof. Dr. S. Ma
Shanghai Key Laboratory of Green Chemistry and Chemical Process
Department of Chemistry, East China Normal University
3663 North Zhongshan Road, Shanghai 200062 (P. R. China)

[c] Prof. Dr. G. Jia
Department of Chemistry
The Hong Kong University of Science and Technology
Clear Water Bay, Kowloon, Hong Kong (P.R. China)

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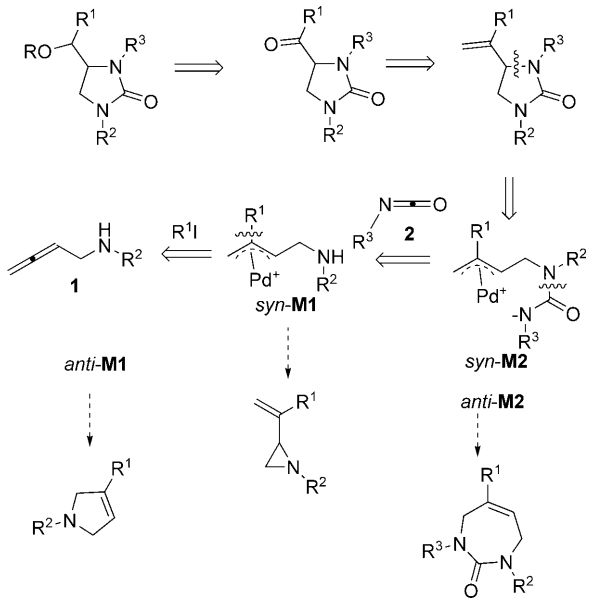
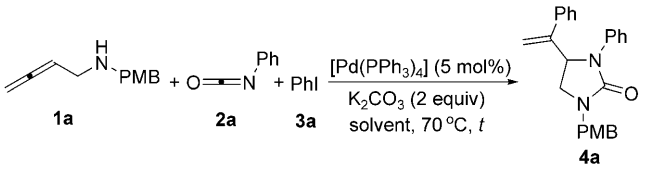


Scheme 2. Reaction scheme.

Table 1. Solvent effect on the palladium-catalyzed reaction of 2,3-allenyl amine **1a** with isocyanate **2a** and iodobenzene (**3a**).[a]

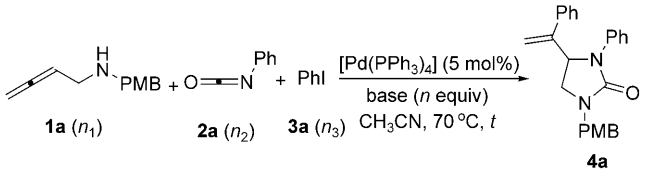


Entry	Solvent	t [h]	Yield of **4a** [%][b]
1	THF	22	72
2	DME	11	70
3	DMF	13	87
4	DMSO	13	76
5	DCE	26.5	77
6	CH_3CN	26.5	96
7[c]	CH_3CN	12	92
8	anisole	11	83
9	toluene	13	54

[a] The reaction was carried out using **1a** (0.2 mmol), phenyl isocyanate **2a** (0.3 mmol), PhI **3a** (0.3 mmol), $[Pd(PPh_3)_4]$ (5 mol%), and K_2CO_3 (2 equiv) in the indicated solvent (4 mL) in a Schlenk tube. [b] Yield of isolated product. [c] $[Pd_2(dba)_3]\cdot CHCl_3$ (2.5 mol%) and PPh_3 (10 mol%) were used. DCE = dichloroethane, DME = 1,2-dimethoxyethane, DMF = *N*,*N*-dimethylformamide, DMSO = dimethyl sulfoxide.

carbonate (1.5 equiv), the yield dropped from 96% to 83% (Table 2, entries 4 and 5). By varying the ratio of the starting materials, we found that **1a**/**2a**/**3a** (1:1.5:1.5) gave the best results. Thus, we defined the reaction of **1a** (1 equiv), **2a** (1.5 equiv), and **3a** (1.5 equiv) catalyzed by $[Pd(PPh_3)_4]$ (5 mol%) and K_2CO_3 (2 equiv) in CH_3CN at 70°C as the standard for further study. With the optimized protocol in hand, we turned to demonstrate the generality of this reaction with a broad range of 2,3-allenyl amines **1**, isocyanates **2**, and organic halides **3**.

Table 2. Further optimization on the palladium-catalyzed reaction of 2,3-allenyl amine **1a** with isocyanate **2a** and iodobenzene (**3a**).[a]



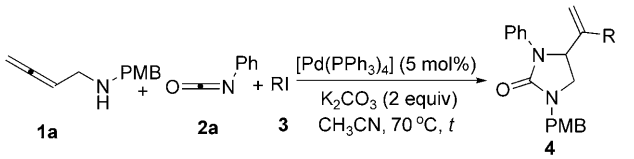
Entry	Base (n [equiv])	n_1:n_2:n_3	t [h]	Yield of **4a** [%][b]
1	Cs_2CO_3 (2)	1:1.5:1.5	11	82
2	Na_2CO_3 (2)	1:1.5:1.5	11	57
3	$K_3PO_4\cdot 3H_2O$ (2)	1:1.5:1.5	11	75
4	K_2CO_3 (2)	1:1.5:1.5	26.5	96
5	K_2CO_3 (1.5)	1:1.5:1.5	12	83
6	K_2CO_3 (2)	1:1.2:1.5	10	83
7	K_2CO_3 (2)	1:2:1.5	10	61
8	K_2CO_3 (2)	1:1.5:1.2	16	73
9	K_2CO_3 (2)	1:1.5:2	16	75

[a] The reaction was carried out using **1a** (0.2 mmol), phenyl isocyanate **2a** (n_2 equiv), PhI **3a** (n_3 equiv), $[Pd(PPh_3)_4]$ (5 mol%) and base (n equiv) in CH_3CN (4 mL) in a Schlenk tube. [b] Yield of isolated product.

As listed in Table 3, phenyl iodides substituted with phenyl, electron-donating, and electron-withdrawing groups are all suitable for this reaction, thus affording corresponding imidazolidinones in good to excellent yields (Table 3, entries 1–10).

This chemistry shows excellent selectivity of the C–I bond over the C–Br bond (Table 3, entry 9). Polysubstituted phenyl iodides also worked smoothly (Table 3, entries 12–

Table 3. Palladium-catalyzed reaction of 2,3-allenyl amine **1a** and **2a** with various organic halides **3**.[a]

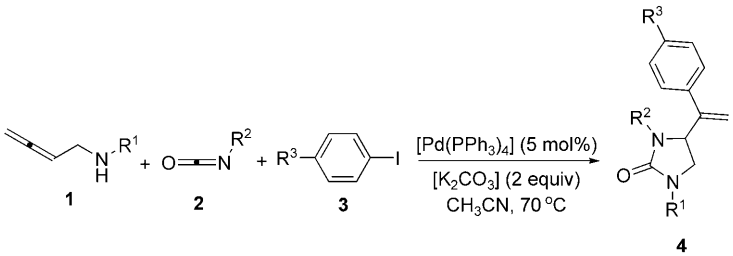


Entry	R (**3**)	t [h]	Yield of **4** [%][b]
1	C_6H_5 (**3a**)	26.5	96 (**4a**)
2	4-$MeO_2CC_6H_4$ (**3b**)	16	90 (**4b**)
3	4-$MeOCC_6H_4$ (**3c**)	16	93 (**4c**)
4	3-MeC_6H_4 (**3d**)	12	83 (**4d**)
5	4-MeC_6H_4 (**3e**)	10	90 (**4e**)
6	4-$MeOC_6H_4$ (**3f**)	16	74 (**4f**)
7	4-PhC_6H_4 (**3g**)	16	92 (**4g**)
8	4-CNC_6H_4 (**3h**)	12	75 (**4h**)
9	4-(4-BrC_6H_4)C_6H_4 (**3i**)	16	86 (**4i**)
10	4-*i*-PrC_6H_4 (**3j**)	17	87 (**4j**)
11	3-thienyl (**3k**)	17	64 (**4k**)
12	3,5-$Me_2C_6H_3$ (**3l**)	17	81 (**4l**)
13	3,4-$Me_2C_6H_3$ (**3m**)	15.5	84 (**4m**)
14	3,4-$(OCH_2)_2C_6H_3$ (**3n**)	15.5	82 (**4n**)
15	(*E*)-C_6H_{13}CH=CH (**3o**)	15.5	70 (**4o**)
16	(*E*)-MeO_2CCH=CH (**3p**)	16	77 (**4p**)
17	4-$NH_2C_6H_4$ (**3q**)	11	63 (**4q**)

[a] The reaction was carried out using **1a** (0.2 mmol), **2a** (0.3 mmol), and **3** (0.3 mmol) in CH_3CN (4 mL) and catalyzed by $[Pd(PPh_3)_4]$ (5 mol%). [b] Yield of isolated product.

14). Furthermore, the reaction of 1-alkenyl iodides also proceeded smoothly to afford the corresponding products in good yields (Table 3, entries 15 and 16). In the case of heterocyclic aromatic iodide **3k**, **4k** was obtained in 64% yield (Table 3, entry 11). It is noteworthy that even 4-iodoaniline with a free amine group is compatible in this process, and gave the substituted **4q** in 63% yield with the aniline functionality untouched for further elaboration (Table 3, entry 17).

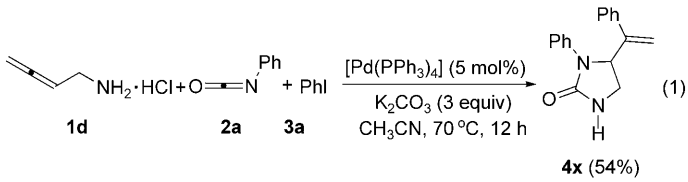
In addition to **1a** and **2a**, *N*-benzyl or *n*-butyl-protected 2,3-allenyl amines and even free 2,3-allenyl amine **1d** can be used for this process (Scheme 3 and Equation (1)): Various



R^1 = Bn, R^2 = Ph, R^3 = Me, 77% (**4r**)
R^1 = Bn, R^2 = Bn, R^3 = Me, 58% (**4s**)
R^1 = *n*Bu, R^2 = Ph, R^3 = Me, 67% (**4t**)
R^1 = Bn, R^2 = 4-$MeOC_6H_4$, R^3 = Me, 75% (**4u**)
R^1 = Bn, R^2 = 4-$NO_2C_6H_4$, R^3 = Me, 83% (**4v**)
R^1 = Bn, R^2 = 4-ClC_6H_4, R^3 = Me, 81% (**4w**)

Scheme 3. The effect of different protecting groups on the nitrogen atoms.

isocyanates (**2**) containing phenyl groups with electron-donating or electron-withdrawing substituent are all suitable substrates for this transformation. The structure of **4** was further confirmed by X-ray diffraction analysis of **4r** (Figure 1).[13]



In conclusion, we have demonstrated a novel three-component cascade reaction of 2,3-allenyl amines with isocyanates and organic halides, which provide an efficient route for the diversified synthesis of polysubstituted imidazolidinones with biological potentials. This method offers several advantages such as good functional-group tolerance, mild reaction conditions, high yields, and easily accessible starting materials (diversity), which will be of board interest for medicinal chemistry. In this transformation, simultaneously, one C–C bond and two C–N bonds are efficiently formed with the breaking of two π bonds. Further work will be dedicated to study the potential applications of these products as well as the development of asymmetric version of this reaction.

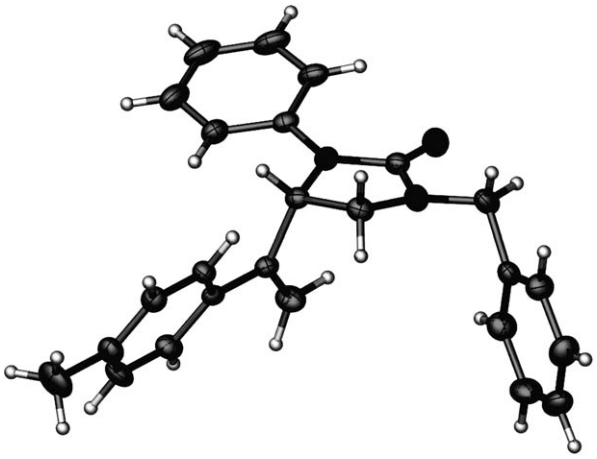


Figure 1. ORTEP representation of **4r**. Ellipsoids set at 30% probability, hydrogen atoms omitted for clarity.

Experimental Section

Typical procedure for the synthesis of 1-(4-methoxybenzyl)-3-phenyl-4-(1-phenylvinyl)imidazolidin-2-one (4a): Once the Schlenk tube containing K_2CO_3 (55 mg, 0.40 mmol) was flamed, dried, and filled with argon, $[Pd(PPh_3)_4]$ (12 mg, 0.010 mmol), **1a** (37 mg, 0.20 mmol), **3a** (58 mg, 0.28 mmol), **2a** (36 mg, 0.30 mmol), and CH_3CN (4 mL) were added sequentially. The resulting solution was heated to and stirred at 70 °C. When the reaction was completed as monitored by TLC, the solvent was evaporated under reduced pressure, and the residue was purified by chromatography on silica gel (eluent: petroleum ether/ethyl acetate = 7:1) to afford 72 mg (96%) of **4a** as an oil: ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.64–7.60 (m, 2H), 7.36–7.29 (m, 7H), 7.18–7.15 (m, 2H), 7.07–7.02 (m, 1H), 6.86–6.82 (m, 2H), 5.44 (s, 1H), 5.26 (s, 1H), 5.09 (dd, J = 9.6, 4.4 Hz, 1H), 4.45 (d, J = 15.0 Hz, 1H), 4.39 (d, J = 15.0 Hz, 1H), 3.79 (s, 3H), 3.60 (dd, J = 9.2, 8.8 Hz, 1H), 3.06 ppm (dd, J = 8.8, 4.4 Hz, 1H); ^{13}C NMR (100.5 MHz, $CDCl_3$) δ = 158.9, 157.6, 145.0, 139.5, 138.0, 129.3, 128.62, 128.55, 128.1, 126.4, 122.5, 118.7, 114.4, 113.9, 56.3, 55.2, 48.0, 47.0 ppm; IR (neat): $\tilde{\nu}$ = 1704, 1610, 1599, 1512, 1502, 1456, 1437, 1417, 1348, 1247, 1205, 1175, 1151, 1112, 1033 cm^{-1}; MS (EI): *m/z* (%): 385 ([M^++1], 7.88), 384 ([M^+], 27.11), 121 (100); HRMS calcd for $C_{25}H_{24}N_2O_2$ [M^+]: 384.1838; found: 384.1837.

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Keywords: allenes · amines · cyclization · heterocycles · isocyanates · palladium

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[12] For initial examples on the reaction of amines with isocyanates, see: a) N. P. Peet, S. Sunder, R. J. Cregge, *J. Org. Chem.* **1976**, *41*, 2733; b) J. A. Bristol, E. H. Gold, R. G. Lovey, *J. Med. Chem.* **1981**, *24*, 927.

[13] Crystal data for **4r**: $C_{25}H_{24}N_2O$, MW = 368.46, monoclinic; space group $P2_1/c$, final R indices [$I > 2\sigma(I)$], $R1 = 0.0660$, $wR2 = 0.1288$; R indices (all data): $R1 = 0.0689$, $wR2 = 0.1305$; $a = 6.1299(3)$, $b = 7.8000(4)$ Å, $c = 42.2547(19)$ Å, $\beta = 92.2890(10)°$, $V = 2018.72$ (17) Å^3, $T = 296$ (2) K, wavelength: 0.71073 Å, $Z = 4$, reflections collected/unique: 22219/3555 ($R_{int} = 0.0251$); number of observations [$> 2\sigma(I)$] 3345, parameters: 261. CCDC-778290 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

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